THE EFFECT OF HETEROGENEITY ON MATRIX ACIDIZING OF CARBONATE ROCKS

A Thesis

by

RYAN SCOTT KEYS

Submitted to the Office of Graduate Studies of
Texas A&M University
in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

December 2009

Major Subject: Petroleum Engineering

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Approved by:

Chair of Committee,	A. Daniel Hill
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ABSTRACT

The Effect of Heterogeneity on Matrix Acidizing of Carbonate Rocks. (December 2009)

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In matrix acidizing, the goal is to dissolve minerals in the rock to increase well productivity. This is accomplished by injecting an application-specific solution of acid into the formation at a pressure between the pore pressure and fracture pressure. A hydrochloric acid solution is used in carbonate reservoirs, which actually dissolves the calcite rock matrix in the form of conductive channels called wormholes. These wormholes propagate from the wellbore out into the reservoir, bypassing the damaged zone. In matrix acidizing of carbonates, there are four parameters that affect performance: the concentration of calcite present, injection rate of the acid, reaction type, and heterogeneity. Of these parameters, this paper will focus on how rock heterogeneity affects performance. To do this, a coreflood and acidizing apparatus was used to acidize heterogeneous limestone core samples. Rock characterizations and volumetric measurements were considered with the results from these experiments, which made it possible to correlate and quantify the results with rock and volume parameters.

It was found that the core samples with more and larger heterogeneities generally required less acid (measured in pore volumes) to achieve breakthrough, that is, a

wormhole created axially from one end of the core to the other. This value for pore volumes to breakthrough was one to two orders of magnitude less than more homogeneous samples. The general procedure and best practices for acidizing the core samples is also detailed in this thesis. This procedure was followed for preparation, coreflooding, and acidizing for all core samples.

DEDICATION

To my parents, who put me in the position that I am today.

ACKNOWLEDGMENTS

I would like to thank to my advisors, Dr. Hill and Dr. Zhu, who provided the perfect combination of guidance and autonomy for which to complete this research with expedience.

Thanks to Omer Izgec, who was a pleasure to work with and helped acclimate me to this research.

Finally, thanks to Dr. Sun for serving on my committee.

TABLE OF CONTENTS

Page

LIST OF FIGURES

LIST OF TABLES

CHAPTER I

INTRODUCTION

An Overview of Matrix Acidizing

Matrix acidizing has been used for decades to increase well productivity by removing damage (usually caused from drilling) near the wellbore. Among the well stimulation techniques, it is cost-effective but sometimes inconsistent due to reservoir heterogeneity, which will sometimes lead to unpredictable results.[1] It was first used in carbonate reservoirs but its application has been expanded in sandstones.[4]

Acidizing is accomplished by injecting acid at a pressure between the pore pressure and fracture pressure, thereby ensuring the acid will travel radially outward from the wellbore without fracturing the formation. The goal is not to alter the reservoir, but to improve the permeability in the vicinity of the wellbore which has been affected by drilling, completion, and/or production operations. In sandstones, the acid removes damage (improves permeability) in the region just a few inches from the wellbore, primarily by dissolving pore-plugging particles. Hydrofluoric acid (HF) is primarily used in sandstone applications because it dissolves clay, feldspar, and quartz particles.[2]

This thesis follows the style of the *SPE Journal.*

Since acidizing of carbonates is the focus of this paper, most attention will be paid to the parameters that affect acidizing of this type of reservoir rock. In contrast to sandstones, the affected region in carbonates can extend up to several feet.[2] Hydrochloric acid (HCl) is used in acid treatments of carbonate reservoirs for a variety of reasons. It is common, cheap, and most importantly, does not produce a detrimental amount of precipitate when reacted with calcite.[4] The reaction for HCl with calcite is:

$$2HCl + CaCO_3 \Leftrightarrow CaCl_2 + H_2O + CO_2$$

In some carbonate reservoirs, dolomite is present in various concentrations. HCl also dissolves dolomite, and the reaction is:

$$4HCl + CaMg(CO_3)_2 \Leftrightarrow CaCl_2 + MgCl_2 + 2H_2O + 2CO_2O$$

The permeability in the near-wellbore region is enhanced when the acid dissolves the matrix and crates conductive channels called wormholes that propagate radially outward from the wellbore.

Performance of acidizing of carbonates varies greatly depending on the application. The parameters that affect performance in carbonates are

- The concentration of calcite in the matrix,
- The acid injection rate,
- The reaction type (which is a function of acid type and temperature), and
- Heterogeneity.[17]

Of these parameters, the injection rate and reaction type can be controlled. The term "heterogeneity" encompasses varying mineralogies, permeabilities, and porosities within the reservoir. The reaction type can be manipulated by varying the type and concentration of acid used.

The Effect of Heterogeneity on Performance

As discussed above, the heterogeneity of the carbonate sample impacts performance and may severely impact results predicted by models based on research done with more homogeneous samples. The effects of matrix acidizing of carbonates with multiscale (micron to centimeter sized) heterogeneities are not well known and are the purpose of this research. Previous research has concentrated on highly homogeneous rocks that were likely selected to limit variability of the results that large scale heterogeneities may have caused.[1,2,16,17]

One study focused on the effects of small-scale heterogeneities in acidizing. Conclusions from this work included a tendency for the acid to follow the path of least resistance through the higher porosity regions within the core samples, resulting in deeper penetration. It was expected that this behavior would be even more noticeable in rocks with large-scale heterogeneities (vugs). For these reasons, rocks with multi-scale heterogeneities were selected for this research.

Experimental Procedure

This paper focuses more on the process and rationale of the matrix acidizing process and rock selection than on the actual results. Research done in parallel (Omer Izgec's dissertation[9]) with the research and experiments included in this paper discusses and analyzes the results in more detail.

Once appropriate rocks were selected, 4" x 20" cores were cut and prepared for a coreflood and eventual acidizing process in an apparatus. Images and porosity distributions were obtained from CT scans of all the cores. Once permeability was determined during the coreflood stage of the experiment, the acidizing process started. Volumes, rates, and pressures were monitored and recorded. From this data, it was possible to determine the number of pore volumes of acid necessary to achieve breakthrough longitudinally through the core sample. In previous research, a pore volume to breakthrough value of about 1 was determined with more homogeneous rocks. A detailed geological classification of these rocks can be found in Reine Vera's thesis.[15]

Details on the components used in the apparatus can be found in Javier Nevito's thesis.[14]

CHAPTER II

EXPERIMENTAL PROCEDURE

The purpose of this section is to describe in detail the procedure for acidizing a core sample. Since this is best described in various steps, it is best for this section to be organized in the form of instructions, especially since this will be used in subsequent research.

Coring

Once a type of rock has been selected, core samples need to be drilled. There is equipment available in the coring lab on the 3rd floor, but after many failed attempts, it became evident that it was more cost-effective to get a professional to cut the cores. It was also logistically impossible to transport a large boulder into the core lab.

Stephen Kocurek, owner of Kocurek Industries in Caldwell, TX, was able to efficiently and quickly cut cores for this project as well as others in the department. His phone number is 919-575-8333. Mr. Kocurek can also acquire most types of rock. As of December 2008, Mr. Kocurek's prices were $60 for a 1"x20" core and $50 for a 4"x20" core.

Saturation

Saturating the core is necessary for any experiment involving a coreflood apparatus. The objective is to achieve single-phase flow. This is done by removing air occupying the pore space and replacing it with the fluid that will be flowed through the core. With any residual air left in the pore space, flow becomes two-phase, air is compressed, and it becomes difficult to determine the core's permeability.

Core saturation is done in the 3rd floor petrophysics lab. The system consists of:

- A large PVC container that houses the core samples and forms the seal,
- A vacuum pump that will draw air out of the sealed PVC container,
- A control box that controls the vacuum pump and displays the pressure,
- A series of tubes, valves, and fittings that connect the pump, box, and container.

First, fill the PVC container with water to the fill line (a red line on the inside of the container).

There are just 3 items that are needed to work with the saturation system: a strap wrench, an open-ended 7/16" wrench, and a large pipe wrench. To close the PVC container, arrange the strap wrench such that it is applying torque in the counter-clockwise direction. Open the pipe wrench so that it spans the square protrusion on top of the PVC container cap. Apply torque with the pipe wrench in the clockwise direction. The strap wrench will exert and equal and opposite torque in the other direction. Tighten the cap

enough such that no threads are exposed and most of the cap is slightly below the lip of the container.

The next step is to attach the fitting to the top of the container cap. Use the 7/16" wrench to tighten the fitting on the threads. When this is done, make sure the correct valves are open so that air can flow from the container to the vacuum pump but not to the other equipment that other projects may be using.

Flip the large red switch on the front of the control box to the 'on' position. The pump should start making noise and the digital display on the front of the box should show the absolute pressure in psi inside the PVC container. Leave the pump on for at least 24 hours. After 24 hours, the pressure should be below 0.8psi. If it is not, then the PVC container is not adequately sealed. Take the cap back off and apply vacuum grease to the threads. Repeat the procedure until a steady-state pressure of 0.6psi is achieved. Depending on the size of the core samples being saturated, there should be a noticeable drop in the water level inside the container, which indicates water has filled the pore space.

Core Cutting

If the cores being used are 4 inches in diameter and 20 inches long, it is necessary to remove 1 inch off the length of the core sample. 20 inches is too long to work with the coreflood hardware. 19 inches is the maximum allowable core length. If the cores being used are 1 inch in diameter, then this step is not necessary.

To remove 1 inch off the length of a 4 inch diameter core, the large core saw in the coring lab should be used. Position the core sample on the L-shaped holder right in front of the saw blade. Slide the core such that exactly 19 inches will remain after the slice is cut. Do not simply measure 1 inch to cut off of one end of the core; measuring this way does not account for the thickness of the saw blade.

Before any cuts are made, make sure the proper protective gear is worn, which includes pants (no shorts), closed-toed shoes, and eye protection.

First, open the valve on the wall that controls the water flow to the saw. The valve is open when it is positioned parallel to the pipe. Next, open the water valve on top of the saw blade. This is done by turning the wing nut counter-clockwise until water begins flowing from the two small tubes located on each side of the saw blade. The water provides lubrication and coolant.

Make sure the core sample is positioned properly such that 19 inches will remain after the slice is cut. Slide the holder and core close to the saw blade but leave a little room. Turn on the saw by flipping the red switch while also making sure nothing is in contact with the blade. Slowly push the holder and core into the blade until a slice is cut. Pull the core away from the blade and shut off the saw blade. Also close both of the water valves.

The core sample is now ready to be used in the acidizing apparatus. If the core sample is not going to be used immediately, store it in water until it is used to make sure it stays saturated. There is a large trash can in the acidizing lab that is filled with water and can be used to store core samples.

Core Scanning and Porosity

Some research requires imagery of the pore space inside the core samples. Once the cores have been prepared, it is possible to obtain this imagery with the CT Scanner located on the 8th floor. The CT Scanner can provide accurate porosity values. If core imagery is not necessary, then another method for determining porosity needs to be used. Weighing the core before and after saturation is another effective means of determining its effective porosity.

Apparatus Preparation

Refilling the Syringe Pumps

The ISCO syringe pumps need to be filled with hydraulic oil to operate. They should have been filled by the last person to use the apparatus, but it is always necessary to check if they are full. There are two pumps that can be used: a 500 mL pump and a 1000 mL pump. Otherwise, the pumps are identical. The amount of fluid in them can be read off of the digital display in the upper right hand corner.

If oil needs to be added, first make sure there is a sufficient amount of oil in the large beaker that serves as the hydraulic oil reservoir. Add as needed to make sure that no air is sucked into the supply tubes. Next, open the inlet valve on the top of the pump and close the outlet valve opposite the inlet valve. Press the "Refill" button on the pump panel. Specify a flow rate of up to 100 cc/min.

Charging the Brine Accumulator

For the instructions to recharge the accumulators, see Figure 2.1.

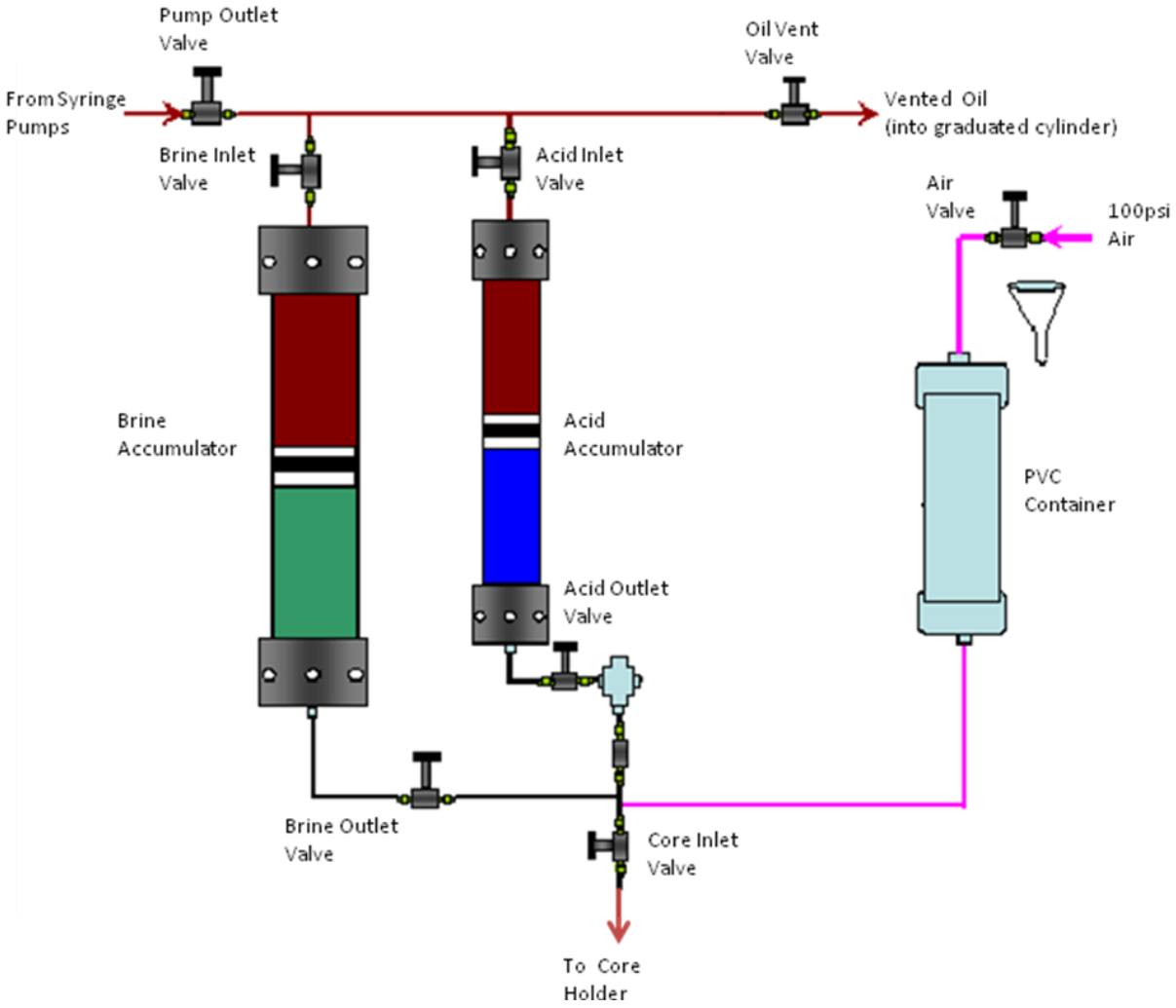

Figure 2.1. Recharging System Schematic

As an experiment is being run, the hydraulic oil is forced from the syringe pumps into the accumulators to move the desired fluid in a 1:1 flow rate ratio (corresponding to a 1:1 piston area ratio). Eventually, the brine supply in the accumulator is exhausted and needs to be refilled to run another experiment.

There should be some idea as to how much brine remains in the accumulator. The previous user of the apparatus should know an approximate amount. If this amount is less than is needed to complete an experiment, brine needs to be added into the brine accumulator. It is prudent to charge the accumulator with more brine than is actually needed to account for any mistakes or unforeseen results. An extra 500 mL is usually enough.

The rationale for the charging system involves using high air pressure to force brine (or another liquid) into the accumulator by manipulating a combination of valves. The first step to refill the brine involves filling the PVC container with brine. For the experiments done in this research, tap water was acceptable. If a particular solution is necessary, mix the solution before filling the PVC container.

Before adding brine, make sure the PVC container is empty. There is a tube with a valve coming off of the bottom of the PVC container (not to be confused with the tube that connects the PVC container with the rest of the charging system). This tube is fairly short and should dump into a container. Open this valve and make to make sure the PVC container is empty. If it is not empty, liquid will come out of the tube and into the

container. This step is very important to ensure that the brine being added is not contaminated by some other liquid.

To fill the PVC container, remove the hydraulic fitting (an adjustable wrench is most useful for this step). Doing this will expose a threaded tube through which the brine can be added. A small funnel is necessary to let the air escape that is being displaced by the brine. Always fill the PVC container with more brine than is necessary to avoid charging the brine accumulator with air. For example, if it is desired to put 1.5 liters of brine in the brine accumulator, fill the PVC container with 2 liters. The extra brine will provide some assurance that air is not being forced into the accumulator.

After filling the PVC container with the desired amount of brine, replace the hydraulic fitting. The next step is to manipulate the valves in the charging system to allow the brine to enter the brine accumulator.

First make sure that ALL valves are closed. For future reference, valves are closed when the arrow on the valve handle is perpendicular to the tubing. When they are open, the arrow is parallel to the tubing. To refill the brine accumulator, the valves (refer to Fig 2.1) should be in the following configuration (Table 2.1):

Table 2.1. Valve Configuration to Charge Brine Accumulator

Valve	**Open/Closed**
Pump Outlet Valve	Closed
Brine Inlet Valve	Open
Brine Outlet Valve	Open
Acid Inlet Valve	Closed
Acid Outlet Valve	Closed
Oil Vent Valve	Open
Air Valve	Open
Core Inlet Valve	Closed

The order in which the valves are opened is unimportant. However, to avoid any mistakes, make sure the Brine Outlet Valve is the last one to be opened. Check the hydraulic fitting on top of the PVC container to make sure it is not leaking air.

After opening the Brine Outlet Valve, hydraulic oil should start coming out of the oil vent tube and into the graduated cylinder. Again, since the brine accumulator has a 1:1 flow ratio, the volume of hydraulic oil in the graduated cylinder equals the volume of brine charged in the brine accumulator. The flow rate of hydraulic oil into the graduated cylinder (and therefore, brine into the accumulator) is approximately 1 liter per hour. Charge the accumulator until the desired volume of brine has been injected. In other words, if it is desired to charge 1.5 liters of brine, then there should be 1.5 liters of

hydraulic oil in the graduated cylinder. Once the desired volume has been charged, close all the valves. Finally, open the valve on the bottom of the PVC container to drain all the remaining brine.

Charging the Acid Accumulator

The process and rationale for charging the acid accumulator is similar to that of charging the brine accumulator. The main difference is the valve configuration that will allow acid to flow into the acid accumulator.

Since acid is a volatile, dangerous substance, the proper precautions are necessary when preparing the desired acid solution. Before doing anything with the acid, make sure long pants, closed-toed shoes, a lab coat, and eye protection are worn.

Also be sure that the acid solution is prepared with the correct diluting liquid. In the experiments for this research, regular tap water was acceptable, so the acid solution was prepared with tap water. However, some acidizing experiments require distilled or purified water, for example. If that is the case, prepare the acid with the same type of liquid that was charged into the brine accumulator.

The acid concentration for this research was 15% HCl by volume. This concentration was chosen because previous research on acidizing of carbonates used the same concentration, which provides a better basis for comparison.

Available commercial concentrations range from 30% to 40% by volume of HCl. Therefore, it is necessary to dilute the acid to achieve the desired concentration. Since the concentrations being discussed are by volume and not weight, only volume ratios need to be considered. Consider the following case if 500 mL of 15% by volume HCl is desired:

In 500 mL of 15% HCl, there is a volume of 75 mL of pure HCl present (500 mL * 0.15 = 75 mL). To achieve this amount of pure HCl, simply calculate how much of the commercial HCl is necessary. For a commercial concentration of 35%, it is necessary to use 214 mL of commercial HCl (75 mL / 0.35 = 214 mL). Simply subtract this amount from the desired amount of 15% HCl to determine the amount of water to mix in the acid solution (500 mL - 214 mL = 286 mL).

Therefore, to make 500 mL of 15% HCl with a commercial concentration of 35% HCl, it is necessary to mix 214 mL of commercial acid with 286 mL of water.

After the acid solution is prepared, it is necessary to charge the acid accumulator with it. Follow the same steps discussed in the preceding section for charging the brine, the only difference being the valve configuration. To charge the acid accumulator with acid, the valves should be in the following configuration (Table 2.2):

Table 2.2. Valve Configuration to Charge Acid Accumulator

Valve	**Open/Closed**
Pump Outlet Valve	Closed
Brine Inlet Valve	Closed
Brine Outlet Valve	Closed
Acid Inlet Valve	Open
Acid Outlet Valve	Open
Oil Vent Valve	Open
Air Valve	Open
Core Inlet Valve	Closed

Again, follow the same procedure used above for charging the brine accumulator. Open the Acid Outlet Valve last.

Since acid is very corrosive, it is necessary to remove any residual acid that may be in the PVC container. After draining the leftover acid, refill the PVC container with tap water. Once the tap water is drained, it is less likely that any components will be damaged from the residual aci

Core Holder Assemblies

Core Holder Sub-Assembly

The core holder sub-assembly should usually already be assembled. The only times it should be disassembled is when one of the components is damaged and needs to be replaced. The core holder assembly is composed of the following components:

- 1 core holder
- 1 rubber sleeve
- 2 ferrules
- 2 Viton 113-70 duro o-rings
- 2 large hose clamps
- 2 mandrels

There are two tapped holes on the core holder. One is filled with a plug, and the other is filled with a hydraulic fitting connected to a valve. The purpose of the valve will be explained later.

First, install one o-ring on each of the ferrules. The ferrules, which are made of hastelloy and are essentially metal rings, have o-ring glands for which to place the o-rings.

Install one ferrule on one end of the rubber sleeve. Lubricate the outside of the ferrule with 15w-40 motor oil to aid installation. Make sure the tapered part of the ferrule slides

into the inside of the rubber sleeve. The motor oil is also used to apply an overburden pressure during an experiment.

With the ferrule on one end of the rubber sleeve, slide the other end of the sleeve into the core holder until the ferrule seats completely. It will be necessary to tap the ferrule with a rubber mallet to get it to seat. Next, flip the core holder over such that the side without the ferrule is facing up. Again, lubricate the inside of the sleeve with motor oil. Slide the other ferrule into the sleeve exactly like the other one. Again, tap the ferrule so that it seats completely on the core holder.

Next it is necessary to install and align the mandrels. The mandrels are components that conform to the outside surface of the core holder and have protruding cylinders that will be used to fasten the assembly onto the core holder stand. Slide the hose clamps over each end of the core holder. Place the mandrels approximately in the middle of the core holder and opposite each other. Slide the hose clamps over the mandrels to hold them in place. There should be red lines on the core holder indicating where the mandrels should be placed. Align the edges of the mandrels with the red lines. Make sure the cylinders are facing in opposite directions (that is, exactly 180 degrees from each other). When the mandrels are in position, tighten very forcefully the hose clamps to keep the mandrels in place. The hose clamps have to be tight enough so that the mandrels support the whole wait of the core holder full assembly.

Core Holder Full Assembly

The core holder full assembly is made up of the following components:

- 1 core holder sub-assembly
- 1 prepared core sample
- 2 platens
- 2 end caps
- 1 floating end platen
- 1 stationary end platen
- 1 tightening assembly

First, place the core holder sub-assembly onto the core holder stand on the apparatus. If it is preferred, there is a mobile core holder stand that will adequately hold the assembly as well. Place the sub-assembly onto the stand such that the mandrels rest on the semi-circular holders. There are two semi-circular pieces that complement the semi-circular holders. Place one over each mandrel and locate the small hose clamps. Slide the small hose clamps over the enclosed mandrels and tighten them. This step will ensure that the core holder assembly will not move on the stand but will allow the core holder to rotate.

With the core holder sub-assembly in place and fastened to the stand, slide the core sample into the middle of the core holder sub-assembly so that it is resting completely on the rubber sleeve. Make sure the core holder is positioned horizontally.

Locate the end cap that goes with the stationary platen. This particular platen is the smaller of the two, has two tubes coming out of its top, and has flanges around its circumference. The primary purpose of the flanges is to keep the ferrule in place inside the core holder. The end cap that goes with it has matching flanges one end of its inside diameter. Slide the platen (flat side first) into the core holder until the flanges bottom out on the core holder. Next, install its corresponding end cap by tightening it onto the large threads on the core holder (the end caps are essentially large nuts). Make sure the flanges on the end platen and the end cap overlap. If they do not overlap, high pressure may turn the end platen into a high-speed projectile during an experiment.

Next, locate the end cap that goes with the floating platen. The floating platen is the larger of the two, has two tubes coming out the side, and has no flanges. This time, the end cap needs to be installed before the platen. Just like on the other side, screw the end cap onto the end of the core holder until it bottoms out on the end of the core holder. Again, the purpose of the end cap is to keep the ferrule in place. Next, slide the floating end platen through the end cap and into the core holder until it bottoms out on the core sample.

The next step is to install the tightening assembly. The tightening assembly should already be assembled, and is made up of a fixture, an adjusting bolt, and two adjusting nuts. The fixture has a hole on one end through which the adjusting bolt moves. The two nuts are on the adjusting bolt on opposite sides of the fixture. Place the fixture on the end cap corresponding to the floating platen. There is a small flange on the fixture that lines

up with a groove on the end cap. A mallet may be necessary to get the fixture to bottom out on the end cap. Next, tighten the bolts on the nut so that the tapered end of the bolt moves into a small conical relief on the floating platen. This will keep the bolt aligned with the rest of the assembly. Tightening the bolt on its fixture will move the floating platen (and therefore, the core sample) further inside the core holder. Keep tightening until the core bottoms out on the inside of the stationary platen, that is, when the bolt becomes very difficult to tighten any further.

The core holder full assembly is now completely assembled. Rotate the assembly on the stand so that the stationary platen is facing up. This vertical orientation will prevent any possible gravity effects during an experiment.

Apparatus Operation

Flow Lines

By this point, the following should have already taken place:

- Core preparation (saturation, cutting, etc)
- Syringe pumps filled with hydraulic oil
- Charged brine and acid accumulator with the appropriate solutions and volumes
- Completely assembled core holder full assembly

The next step is to connect the appropriate lines from the apparatus to the core holder assembly via the tubes coming out of the platens. The side of the core holder assembly

with the stationary platen (should be facing up) is the inlet. Consequently, the side with the floating platen (facing down with tubes sticking out horizontally) is the outlet.

Connect the lines coming out of the apparatus to the platens in the following configuration (Table 2.3). The lines should be clearly labeled. Again, the core holder inlet (top) is composed of the two lines coming out of the stationary platen. The outlet (bottom) is composed of the two lines coming out of the floating platen.

Table 2.3. Apparatus Line Configuration

Line	**Connect to:**
Acid Inlet	Inlet
Pressure Transducer Inlet	Inlet
Backpressure	Outlet
Pressure Transducer Outlet	Outlet

The two tubes in each platen flow to identical locations, so there is no need to differentiate between them. The lines connect to the tubes with hydraulic fittings. The fittings are to be tightened onto the threads with a 7/16 inch wrench.

Applying Overburden Pressure

Overburden (or 'surrounding') pressure is applied to the small volume between the inside of the core holder and the outside of the rubber sleeve. The o-rings and ferrules ensure that the pressurized motor oil stays in this volume.

The reason for the overburden pressure is to ensure that all liquid flows through the core sample. The pressure applied to the outside of the rubber sleeve "squeezes" the core sample, preventing any fluid from going around its circumference. Consequently, this overburden pressure must at all times be greater than the inlet pressure. If it is not, fluid can bypass the core sample, and in some cases bypass the ferrul and enter the space between the sleeve and core holder. Therefore, it is absolutely necessary for the overburden pressure to be AT LEAST 300psi greater than the inlet pressure.

There is one line left over after connecting the other lines to the core holder. This line is labeled "Overburden" and should be connected to the fitting located on the side of the core holder. The fitting is connected to a valve, which in turn is connected to the core holder. The valve is installed on a hole in the core holder that provides a conduit for the motor oil to pass through into the aforementioned small volume outside of the rubber sleeve. Open the valve before installing the line.

There is an Enerpac hand pump used to supply the overburden pressure. Make sure it is filled with 15w-40 motor oil. On the front panel of the apparatus there is a valve next to a pressure gauge labeled "overburden." Make sure this valve is open. Begin pumping the motor oil with the Enerpac pump until a pressure of about 700psi is achieved. Monitor the overburden pressure and make sure it remains constant. If it drops, there is a leak somewhere, in which case the lines and fittings should be checked for leaks. If there is no external leak, it is likely that the ferrul did not seal properly with the rubber sleeve, and the core holder assembly needs to be disassembled to ensure a proper seal.

It is important to monitor the overburden pressure at all times. If there is a sudden drop, then the experiment needs to be stopped. During an experiment, make sure the overburden pressure is always at least 300psi greater than the inlet pressure. It will be necessary to operate the Enerpac pump during an experiment to meet this requirement.

Data Acquisition System

This step is to be done sequentially after the overburden pressure is applied and before the syringe pump is turned on.

The pressure drop across the core sample at any given time is monitored by three differential pressure transducers that operate at various ranges. These transducers send signals to a data acquisition system, which in turn is read by the computer. Labview records and compiles the data as a function of time.

Open up the appropriate LabView file from the C drive on the computer. Once it is open, click the "Window" menu and click "Block Diagram." Another window shows up which shows the schematic of the particular LabView program. Double-click the "Write LabView Mesurement File" box in the schematic. A window shows up that prompts the user for a directory and file name for which to write the data. Enter the name of the experiment in this window and press "OK." This step has to be done for every experiment, otherwise LabView will write over the previous experiment's data, which will be lost.

Click back over to the LabView Front Panel window and enter the appropriate data for core geometry (length and diameter), viscosity, and flow rate. When it is run, the program will show a graph of the pressure drop versus time. With the other entered data, the program then calculates the core's instantaneous permeability based on the pressure drop.

Determining Core Permeability

At this point, it is possible to turn on the syringe pumps and to begin flowing brine through the core sample to determine permeability.

To calculate permeability accurately, the pressure drop across the core needs to be in a certain range. If the pressure drop is too low, then small fluctuations in pressure will cause a relatively large margin of error for the calculated permeability. If the pressure drop is too large, it is impossible to apply backpressure because the inlet pressure will increase beyond the mechanical limit of the core sample and hardware. Therefore, calculate a flow rate such that the pressure drop is somewhere between 100psi and 1000 psi. There should be some range or estimate as to what the core permeability should be. Use this estimate to calculate the flow rate. The equation to be used to calculate the desired flow rate is Darcy's linear flow equation.

To begin injecting brine, refer again to the schematic of the charging system (see Figure 2.1). There is no order for opening and closing the valves in this step. The valves should be in the following configuration (Table 2.4):

Table 2.4: Valve Configuration for Brine Injection

Valve	**Open/Closed**
Pump Outlet Valve	Open
Brine Inlet Valve	Open
Brine Outlet Valve	Open
Acid Inlet Valve	Open
Acid Outlet Valve	Closed
Oil Vent Valve	Closed
Air Valve	Closed
Core Inlet Valve	Open

To better understand what is accomplished with this configuration, essentially what is happening is the syringe pump pumps hydraulic oil, which moves the piston inside the brine accumulator, which moves an equal amount of water through the pump. With the appropriate valve configuration, there is nowhere else for the hydraulic oil and water to go than through this path. The Acid Inlet Valve should be open in this case to prevent any unnecessary pressure fluctuations when the acidizing step begins.

Before starting the syringe pump, again make sure the overburden pressure is at least 700psi. Always remember to keep the overburden pressure greater than the inlet pressure, and to add pressure accordingly during the experiment.

To turn on the syringe pump, follow these steps:

1. Make sure the valve between the hydraulic oil reservoir and the refill pump is closed. If it is not, the pump will not move oil into the accumulator.
2. Press the "Flow Rate" button.
3. Enter the desired flow rate in cc/min and press "Enter"
4. Press "Run"

The digital display should show the flow rate, the pump pressure, and the volume of hydraulic oil left. It is important to monitor the volume during the course of the experiment.

After a few seconds of running, the pump pressure begins to build, which indicates that hydraulic oil is moving into the accumulator and beginning to force brine through the core. It usually takes at least 100 cc of oil before the core inlet pressure climbs to a non-negligible amount. The core inlet pressure can be monitored on the pressure gauge labeled "inlet pressure" located on the front panel of the apparatus. Once again, make sure the overburden pressure is always at least 300psi greater than this inlet pressure.

The inlet pressure will increase relatively dramatically at first, and the rate of increase will slow as the pressure drop asymptotically approaches the steady-state pressure drop for the entered flow rate. At some point before this steady-state pressure drop is reached, water will begin flowing out of the outlet line into the container. The inlet pressure (which is equal to the pressure drop since the outlet pressure is atmospheric pressure) can be monitored in 3 places: the display on the syringe pump, the pressure gauge for the inlet pressure, and the digital readouts on the pressure transducers. Of these, the pressure transducers provide the most precise pressure measurement.

When the pressure drop becomes fairly constant (that is, with negligible fluctuation), write down this pressure drop and permeability. After the backpressure is applied (next step), the pressure drop should be the same. Check for leaks and repair as needed.

Applying Backpressure

A backpressure is necessary to simulate down-hole conditions. One of the by-products of the chemical reaction between calcite and HCl is gaseous carbon dioxide. It is desirable to keep the carbon dioxide in solution to eliminate effects of two-phase flow. At room temperature, a water-carbon dioxide mixture is a single phase liquid above 890 psi[11]. The minimum pressure inside the core sample (at the outlet) needs to be above this pressure.

A backpressure regulator is used to keep the outlet pressure at a specified level. An external pressure is applied to the backpressure regulator, which exerts the resistance

upstream, or in this case, to the core outlet. The resistance is the same as the external pressure applied to the regulator. That is, the backpressure regulator maintains the same pressure upstream based on the applied external pressure. When the outlet pressure reaches the desired backpressure, the regulator allows the flow of fluid through it. According to the manufacturer, the backpressure regulator will not work for flow rates lower than 4 cc/min. Therefore, 4 cc/min is the minimum flow rate for this apparatus.

After the steady-state pressure drop across the core has been determined, it is necessary to apply the backpressure in preparation for the acidizing step. Open the valve for the nitrogen tank and adjust the pressure regulator until a pressure of at least 900psi is achieved. Once the nitrogen pressure begins acting on the backpressure regulator, the flow of liquid coming out of the outlet line will stop momentarily. At first, the pressure drop will drop dramatically as no fluid is being forced across the core. This is due to the time it takes the syringe pump to build up the necessary pressure that is required to overcome both the pressure drop and the backpressure. A simple calculation is useful to determine the maximum inlet pressure at the given flow rate:

Back pressure + Pressure drop = Inlet pressure

In this case, the pressure drop is the same as the one that was calculated before the backpressure was added.

As the inlet pressure approaches this value, brine should begin to flow again out of the outlet line. The steady-state pressure drop observed should be very similar or identical to the one observed before applying the backpressure.

Once this steady-state pressure drop is achieved again, press the "Run/Record" button in LabView. The program will begin recording data points every 5 seconds for the pressure drop, then calculating permeability. There will be graphs showing real-time results for both on the Front Panel window. Record at least 5 minutes of this steady-state pressure drop in Labview before beginning the acidizing process. Recording an extended period at this steady-state pressure will provide evidence that it has been achieved, and will remove any doubt as to what the core's permeability is. Check for leaks and repair as needed.

Maximum Pressure

Some of the core samples cracked during various experiments. In each case when a core sample failed, a high overburden pressure was necessary in order to exceed a high pressure drop. After each of these experiments when high pressure was necessary, there was an audible noise coming from the core holder accompanied by a vibration. Upon removal of the core sample from the core holder, each of these cores was broken in half. From this empirical evidence, it appears that there is a mechanical limit for the core samples when considering the overburden pressure. The lowest overburden pressure for which a core sample failed in this manner was observed to be 2800 psi. Therefore, to

limit the number of failed experiments, a maximum overburden pressure of 2500 psi was implemented.

With this maximum overburden pressure, there is a maximum inlet pressure of 2200 psi due to the strict minimum of a 300 psi pressure difference between the inlet pressure and overburden pressure. With a backpressure of 1000 psi, this limitation imposes a maximum pressure drop across the core of 1200 psi. As discussed in the previous sub-section,

Back pressure + Pressure drop = Inlet pressure.

Or in this case with a maximum inlet pressure of 2200 psi,

Back pressure (1000 psi) + Pressure drop (1200 psi) = Inlet pressure (2200 psi).

Acidizing the Core Sample

With LabView still recording, the next step is to switch from brine to acid. This should be done seamlessly and with the syringe pump still running. This step should be done as quickly as possible to limit the amount of time that both brine and acid are being pumped simultaneously.

In this specific order, do the following:

1. Open the Acid Outlet Valve
2. Close the Brine Outlet Valve
3. Close the Brine Inlet Valve

The valve configuration should be as shown in Table 2.5.

Table 2.5. Valve Configuration for Acid Injection

Valve	**Open/Closed**
Pump Outlet Valve	Open
Brine Inlet Valve	Closed
Brine Outlet Valve	Closed
Acid Inlet Valve	Open
Acid Outlet Valve	Open
Oil Vent Valve	Closed
Air Valve	Closed
Core Inlet Valve	Open

Again, the Acid Inlet Valve should have already been opened during the brine injection step. Keeping this valve open (and the Acid Outlet Valve closed) will prevent any undesirable pressure fluctuations. The goal is to achieve a nearly seamless transition between brine injection and acid injection. Check for leaks and repair as needed.

Once acid begins to flow through the core, it may take a while before the pressure drop across the core begins to decrease. Eventually, the pressure drop (and consequently, the permeability) will drop. Continue to inject acid until a pressure drop of less than 15 psi is achieved, which indicates that a conductive wormhole channel has been created through the length of the core. Another indication of breakthrough is when a gas and fluid mixture begins coming out of the outlet line.

After breakthrough has occurred, press “Stop Recording” in the LabView Front Panel to stop the program. Keep the LabView program open. Also make not of how much acid was injected, which can be done by recording the volume left in the syringe pump just before acid injection started and just after it ended.

Now, relieve the pressure acting on the backpressure regulator. To do this, first close the valve on top of the nitrogen tank to close off the nitrogen source. Next, open the red relief valve that tees off from the line going to the backpressure regulator. Doing this will vent all of the nitrogen that has been in the line and regulator. With the backpressure at zero, the residual effluent in the core sample will instantly become a two-phase mixture of carbon dioxide gas, acid and brine. This mixture will flow quickly out of the outlet line until no more carbon dioxide is present, at which point a single phase acid and brine mixture will flow.

Next, reverse the procedure for switching to acid injection. It is necessary to flow brine through the system to remove the acid present. This will reduce the amount of time the components are exposed to the acid and therefore will reduce corrosive effects.

In this specific order, do the following:

1. Open the Brine Inlet Valve
2. Open the Brine Outlet Valve
3. Close the Acid Outlet Valve
4. Close the Acid Inlet Valve

This will result in the valve configuration for brine injection. Flow several hundred cc's of brine through the system to ensure no acid is present. Afterwards, press the "Stop" button on the syringe pump.

There may be some residual pressure at the inlet valve. To relieve this pressure, open the valve labeled "Bypass" on the front panel of the apparatus. The fluid providing this residual pressure will exit out of the same outlet line into the container. Close the Bypass Valve after no more fluid is left to evacuate.

Post-experiment Procedure

There are a number of things to do after an experiment is completed. They are listed below. Complete these tasks in order.

1. In LabView, click "Save." The saved file can be opened with Microsoft Excel. The data recorded is the pressure drop across the core sample taken every five seconds. In this file, make any pertinent notes and record any observations. Be sure to record the amount of acid injected, the flow rate, the core geometry, and the name of the experiment.
2. Relieve the overburden pressure. To do this, open the valve on the side of the Enerpac pump. The motor oil will flow back into the pump from the core holder assembly.
3. Remove the inlet and outlet lines from the core holder assembly.
4. Remove the overburden pressure line from the core holder. Some residual pressure will remain in the core holder, so some motor oil will slowly drip out. Catch this oil in a container and let it drain for a few minutes. Removing as much of the oil as possible is useful because it will be easier to remove the core sample.
5. Record the approximate amount of brine left in the brine accumulator.
6. Remove any acid left in the acid accumulator. This will prevent any unnecessary corrosion from occurring. To do this, open the bypass valve and both acid accumulator valves. Turn back on the syringe pump until all acid is removed.
7. Disassemble the core holder full assembly, leaving the sub-assembly intact.

8. Remove the core sample and store it in water until it is needed again for either observation or a post-acidizing CT scan.
9. Clean up any messes that may have happened during the experiment
10. Close all the valves.
11. Refill the syringe pump with hydraulic oil.
12. Repair anything that may have been damaged during the experiment.

CHAPTER III

EXPERIMENTAL RESULTS

Rock Selection

Rationale for the rock selection involved finding an appropriate analog or proxy for reservoir rocks found in the Shu'aiba formation, a carbonate formation near the Arabian Peninsula spanning from south of the United Arab Emirates to Iraq (shown in green in Fig. 3.1).

Of particular interest is a gas reservoir within the Shu'aiba just north of Qatar in the Arabian Gulf (shown circled in blue in Fig. 3.2).

The Shu'aiba formation is the topmost layer in the Thamama group of carbonates. A section view of this group can be seen in Fig 3.3.

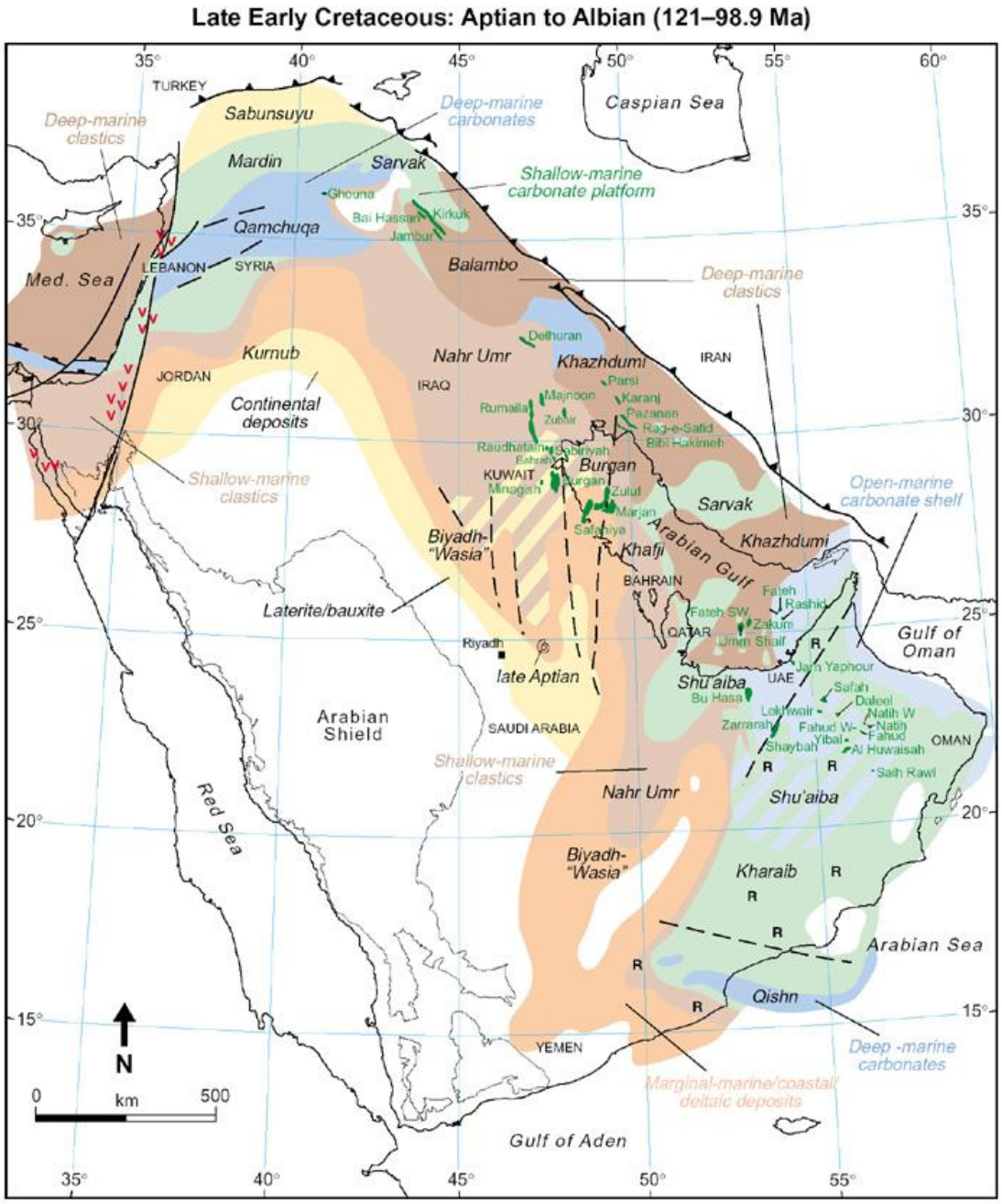

Fig 3.1. Formations in or near the Arabian Peninsula.[13]

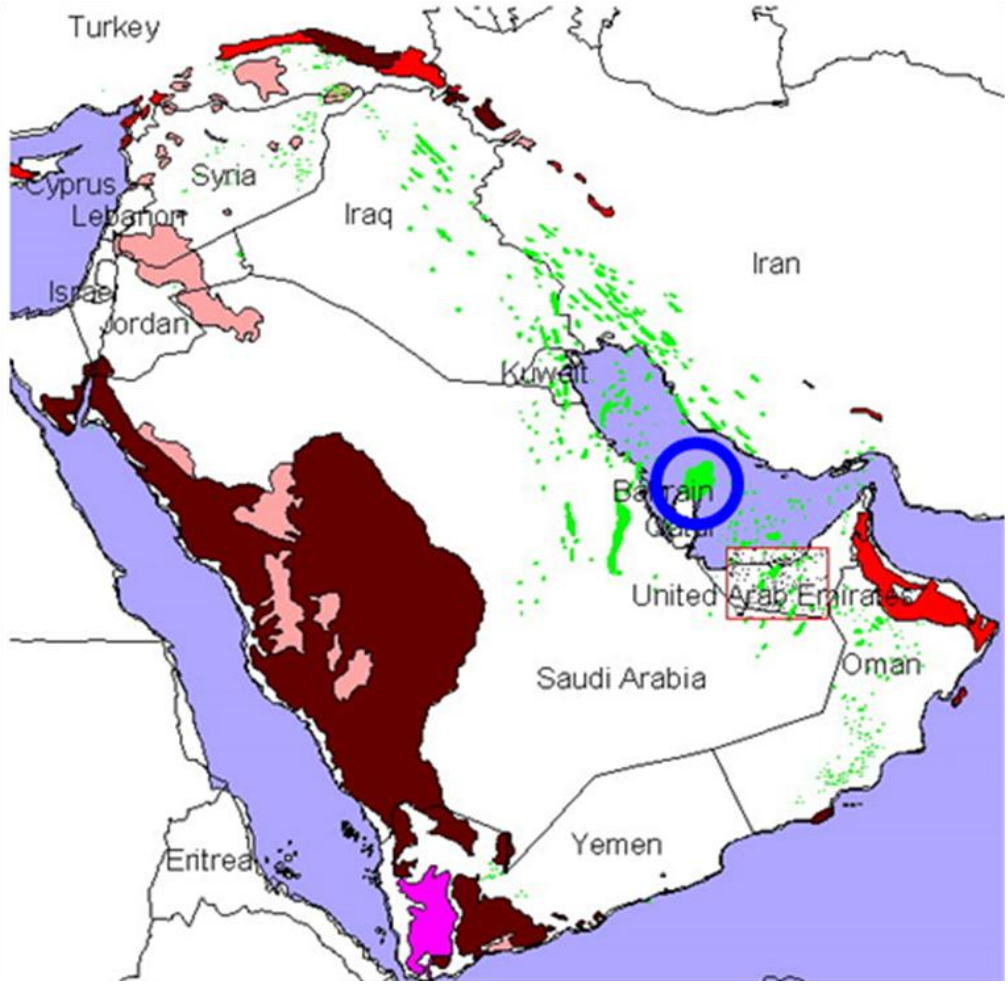

Fig 3.2. Map of Oil and Gas Reservoirs in the Shu'aiba.[13]

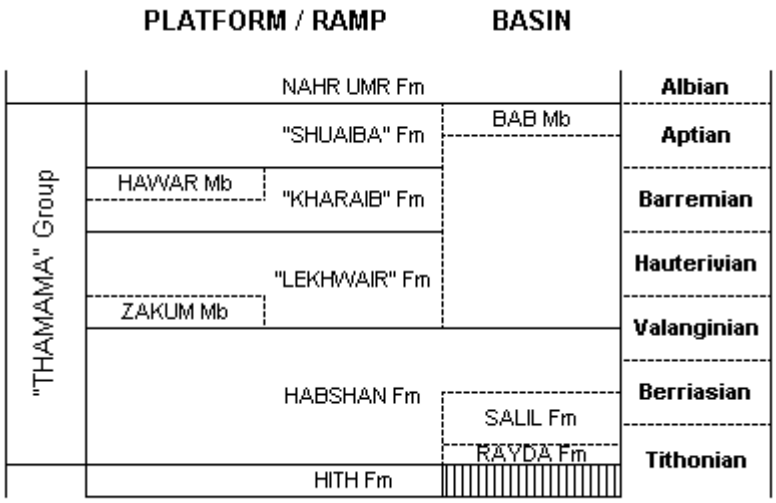

Fig. 3.3. Stratigraphy of the Thamama Group[13].

The Shu'aiba Formation is an Aptian Stage (Early Cretaceous, 125-112 mya) carbonate layer whose deposition occurred in shallow marine environments. Conditions were favorable for deposition of reef limestone. Lithology varies greatly depending on location and depth due to the varying environments that eventually contributed sediment, ranging from chalky lime mudstone to coarse-grained, vugular limestone. Also of note are interspersed rudist beds. Rudists, a type of marine bivalve, thrived during the Aptian stage in these shallow marine environments and can be used as a fingerprint for depositional environment and age[13].

With knowledge of the Shu'aiba formation, it was possible to find an appropriate analog from which samples could be more easily obtained. The Edwards Formation contains limestone that very closely resembles the carbonates found in the Shu'aiba. Deposition of the Edwards also took place in a variety of marine environments in a shallow sea that

once covered much of Texas. Its age ranges from 120 million years for the bottom to 115 million years on the top. The top of the Edwards contains coarse-grained limestone rock that is often highly heterogeneous with a variety of vug sizes and distributions. A one meter thick layer of rudists can be found in the top section of the Edwards formation. Because of these similarities, it was determined that core samples of Edwards Limestone would provide an adequate proxy and would be appropriate for this research[5].

The search for limestone boulders began west of Austin, Texas, where there are abundant Edwards Limestone outcrops near creeks, rivers, and roads. Samples were obtained from two locations:

- The bridge on Hamilton Pool Road crossing the Pedernales River, between the towns of Bee Cave, TX and Cypress Mill, TX.
- A property just south of Barton Creek within the Austin city limits.

A map showing these locations can be seen in Fig. 3.4.

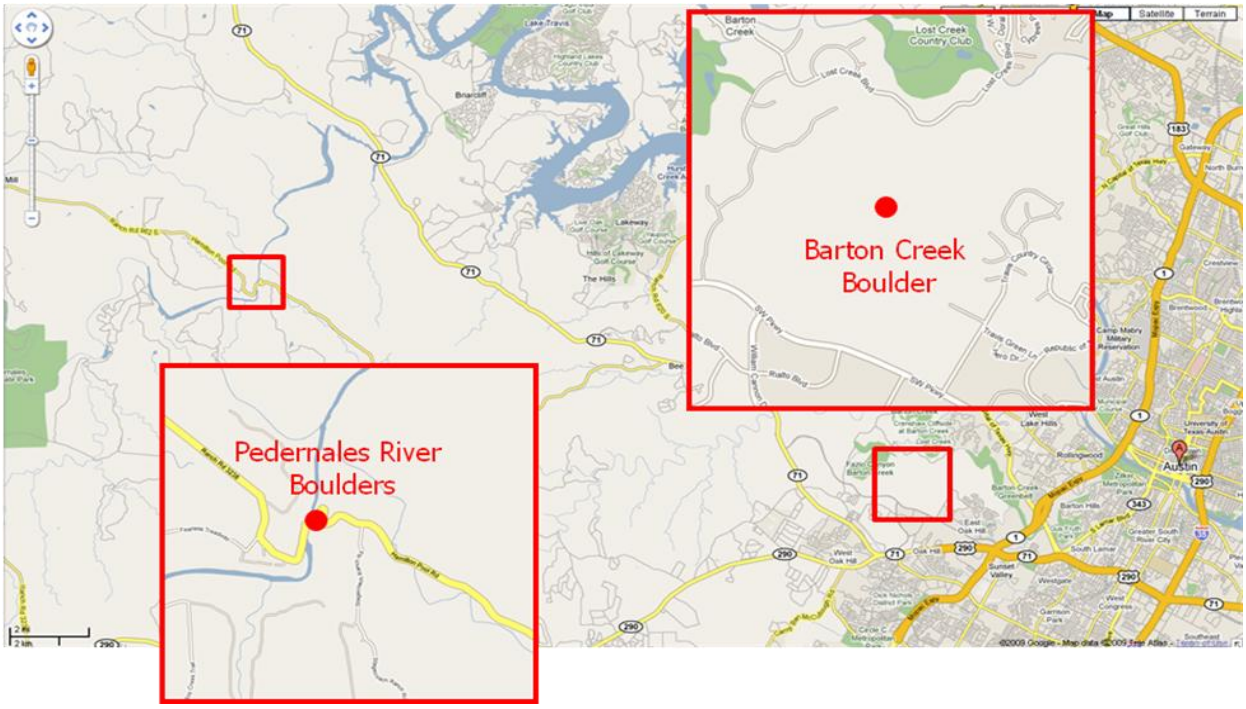

Fig. 3.4. Map of Edwards Limestone Sample Locations.

The Pedernales River samples were found on the eastern bank of the river, likely deposited there during periods of high flow. Because of this, the original location of these boulders could not be determined. A photo of the Pedernales and abundance of boulders was taken from the bridge and can be seen in Fig. 3.5.

Fig. 3.5. Photo of Pedernales Boulders.

These boulders proved to be excellent candidates for acidizing experiments due to the presence of vugs. However, coring was very difficult due to the relatively small, irregular shapes of these boulders. The rocks could not be adequately clamped for use with the coring drill. After several failed attempts, it was determined that a large boulder would need to be found and the cores drilled by a professional.

A large, vuggy limestone boulder was later found at a site located at 6608 Southwest Parkway in Austin, Texas. The land was formerly a working ranch that was bought for development purposes. Therefore, a large amount of excavation was being done to prepare for development. Several large boulders were located near the areas being

excavated, many of which were vuggy Edwards Limestone. The excavation was taking place approximately 500 meters south of Barton Creek, which is known for its outcrops of Edwards Limestone. This boulder (Fig 3.6) was delivered to Kocurek Industries for coring.

Fig. 3.6. Photo of the Barton Creek Boulder.

Barton Creek

The experimental results for the core samples from the boulder named "Barton Creek" are in this section. Each experiment was conducted with the procedure detailed in the "Experimental Procedure" section of this document. Again, the focus of the research discussed in this paper is on the experimental procedure and the data obtained. Research and analysis done in parallel with the results from these experiments is detailed in Omer Izgec's dissertation.

Each experiment's name has a prefix of "Barton Creek" followed by a number suffix that indicates the chronological order in which the experiment was completed. There are gaps in the chronological sequence because some experiments were omitted due to apparatus malfunction, user error, or undesirable core sample properties (e.g. permeability too low).

In this subsection, graphs (Appendix A) showing normalized pressure and permeability as a function of pore volumes of acid injected for each experiment. The pore volume of each core sample was calculated via information from the CT scan. The outputs from the LabView files show permeability and pressure as a function of time, so the time was multiplied by acid flow rate to achieve values for number of pore volumes injected. Pressure values were normalized to the initial (steady-state) pressure. Permeability values were normalized to the greatest permeability achieved, that is, the permeability after breakthrough.

The pressure profile as affected by several parameters. At the acid front, or the end of the developing wormhole, there is a negligible pressure drop between it and the front of the core sample. It is evident that the pressure drop did not decrease linearly with most of these experiments. Further, there are many occasions when the pressure drop does not decrease at all for an extended period. It is likely that during these "plateaus" result from the wormhole propagating radially in the core sample instead of axially. In the case of a straight, axial wormhole with no radial deviation, the pressure drop would be expected to decrease linearly.

There are also many cases when the pressure drop decreases dramatically. When this happens, it is likely that the acid front has arrived at the preferential flow path, that is, the path through the core with the least resistance. This preferential flow consists of the shortest distance between long vugs axially along the core sample. Because there is significantly less matrix to dissolve between these vugs, the acid propagates quickly, and therefore the pressure drop decreases dramatically.

The geological characterizations of these cores were obtained from Riene Vera and can be seen in Appendix B.

Church Road Boulders

A desire for experiments with highly heterogeneous carbonates was identified, so a search for such rocks began. The same source that supplied the Barton Creek boulder was searched, but no highly heterogeneous boulders were found.

Coincidentally, there were several carbonate boulders across the street from Texas A&M at St. Mary's Catholic Center. They appeared to be highly heterogeneous, with very high vugular porosity fractions, so they were delivered to Steven Kocurek for coring.

8 cores were ordered, each having centimeter-scale heterogeneities. They were saturated, scanned, and acidized exactly as the Barton Creek boulders.

Upon attempting to determine the permeability of these core samples, it was found that the pressure drops across the core never stabilized at any flow rate, except for one. Several attempts were made with all 7 of these cores to find a steady-state pressure drop, but in each the pressure drop climbed exponentially until the maximum inlet pressure of 2000psi was reached while flow rates were decreased to 1 cc/min.

For the core sample that did display a steady-state pressure drop, it was evident that the core had a natural interconnected vug network, as the pressure drop was less than 10psi. This core was therefore disregarded since acidizing would not be necessary.

The reason for the constantly increasing pressure is not known. For some reason, the cores were not flowing any water. It was observed that some of the cores were not well consolidated and had and abundance of loose sediment. It is possible that some of this sediment migrated and clogged potential pore throats, thereby preventing the core from flowing water.

The maximum pressure drop was imposed because several of the cores cracked under the 2500psi overburden pressure needed to maintain a pressure drop of 2000psi. Again, due to the presence of unconsolidated matrix, these cores were weaker than the Barton Creek cores, which made them more prone to cracking. Therefore, all efforts to acidize these cores were abandoned.

The geological characterizations of these cores were obtained from Riene Vera and can be seen in Appendix C.

Pre- and Post-acidizing CT Images

This section discussed images (Appendix D) obtained from the CT Scanner before the core sample was acidized. The images show the pore space that is defined as vugs. Also included are images obtained from the CT Scanner after the core sample was acidized. These images show the wormholes created by the acid propagation in addition to the vugular pore space.

Both radial and axial wormhole behavior is evident, which corresponds to the pressure profiles in the previous section. The connection between the wormhole behavior and the individual pressure profiles was established in the previous section as well.

In two of the images, for BC3 and BC6, there are what appear to be large vugs that span the entire cross section of the core. These are a result of the core sample breaking in half during removal. There is no doubt that these breaks occurred after the experiment because there was plenty of evidence to suggest they broke while attempting to remove them. At times, the rubber sleeve did not separate from the core sample despite relieving the overburden pressure. This is probably a result of high applied overburden pressure (close to the maximum) coupled with the presence of large vugs on the circumference of the core sample. The rubber sleeve likely got caught in these large vugs and was unable to separate itself from the core sample surface. The only way to remove the cores at that point was to apply an impulse in an attempt to separate the core from the sleeve. When doing this for BC3 and BC6, the core broke in half.

CHAPTER IV

CONCLUSIONS

In Table 4.1, pertinent data for each experiment is compiled. The acid injection rates are shown for each experiment. The permeabilities were calculated from the steady-state pressure drop during each experiment. The porosity values were calculated by using data from the CT Scanner. In the last column, pore volume to breakthrough (PVbt) values are shown. This value is calculated by dividing the amount of acid injected into each core sample by its pore volumn. The pore volume is calculated by multiplying the bulk volume of each core by its porosity.

Table 4.1. Property Data for Each Core Sample

Name	Flow Rate (cc/min)	Perm (mD)	Porosity (frac)	PVbt
BC1	12	2.5	0.20	0.04
BC2	12	1.5	0.21	0.04
BC3	12	1.3	0.21	0.04
BC4	8	1.7	0.19	0.11
BC5	8	2.2	0.18	0.06
BC6	4	1.9	0.18	0.08
BC7	8	1.8	0.17	0.08
BC9	12	8.5	0.22	0.09
BC10	12	23.3	0.22	0.06
BC11	24	15.0	0.22	0.13

Table 4.1, Continued

Name	Flow Rate (cc/min)	Perm (mD)	Porosity (frac)	PVbt
BC12	24	3.9	0.22	0.12
BC13	12	11	0.22	0.15
BC14	16	17	0.22	0.11
BC15	16	21	0.22	0.15
BC21	20	25.6	0.22	0.08
BC22	13	2.2	0.2	0.08

There were enough experiments to get a porosity-permeability plot. This plot is shown in Fig. 4.1. Despite coming from the same boulder, the permeabilities varied from 1.3 mD to 25.6 mD for the experiments shown. The trend indicates that increasing porosity values yielded higher permeabilities. However, several experiments failed (hence the gaps in the numeric sequence) due to permeabilities too low to carry out an experiment. As mentioned earlier in this paper, the minimum flow rate for the backpressure regulator is 4 cc/min. With this flow rate, inlet pressures for these low-permeability cores would exceed the maximum allowable pressure drop for the system when a backpressure of 1000 psi and a maximum overburden pressure of 2500 psi are considered. With a maximum backpressure of 2500 psi, there is a 2200 psi maximum for the inlet pressure. Again,

Back pressure (1000 psi) + Pressure drop (1200 psi) = Inlet pressure (2200 psi).

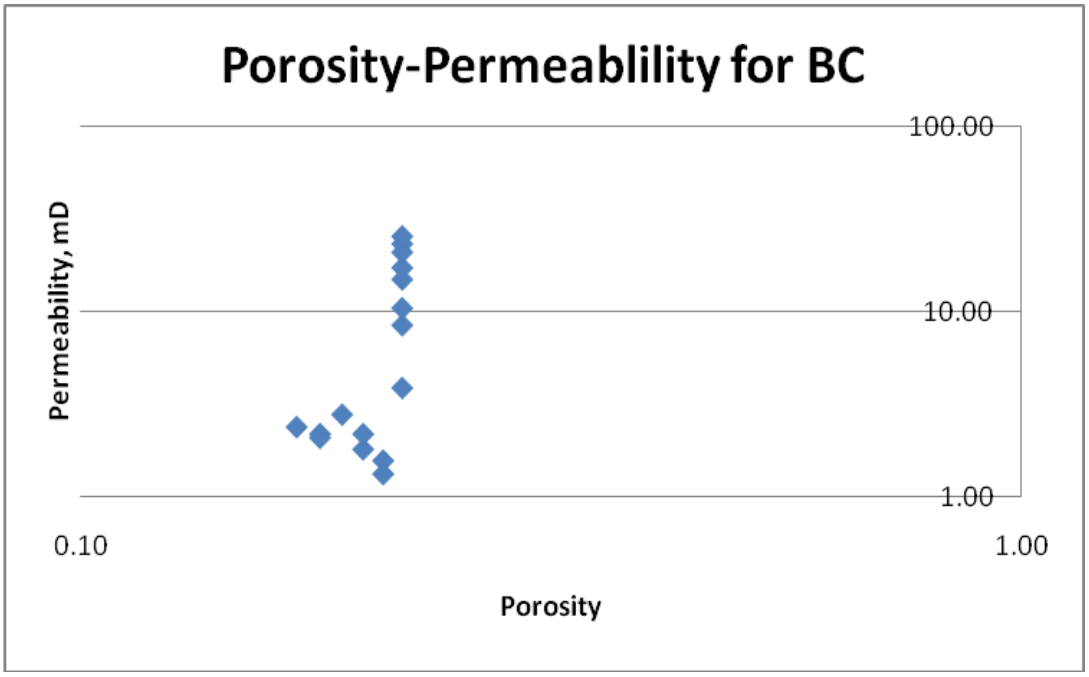

Fig 4.1. Porosity-permeablility Plot for Barton Creek Core Samples.

As discussed in the introduction, carbonate matrix acidizing performance is affected by a variety of parameters including acid injection rate. There should be an optimal injection rate that will maximize wormhole penetration for a given volume of acid injected. In other words, it is desirable to achieve maximum penetration with a minimal amount of acid. One way to measure this relationship is by calculating the amount of acid that is necessary to achieve breakthrough in a core sample, and normalizing that volume of acid by the core's pore volume. This value, known as pore volumes to breakthrough, is affected by injection rate. Fig 4.2 shows a graph showing the PVbt values for different flow rates.

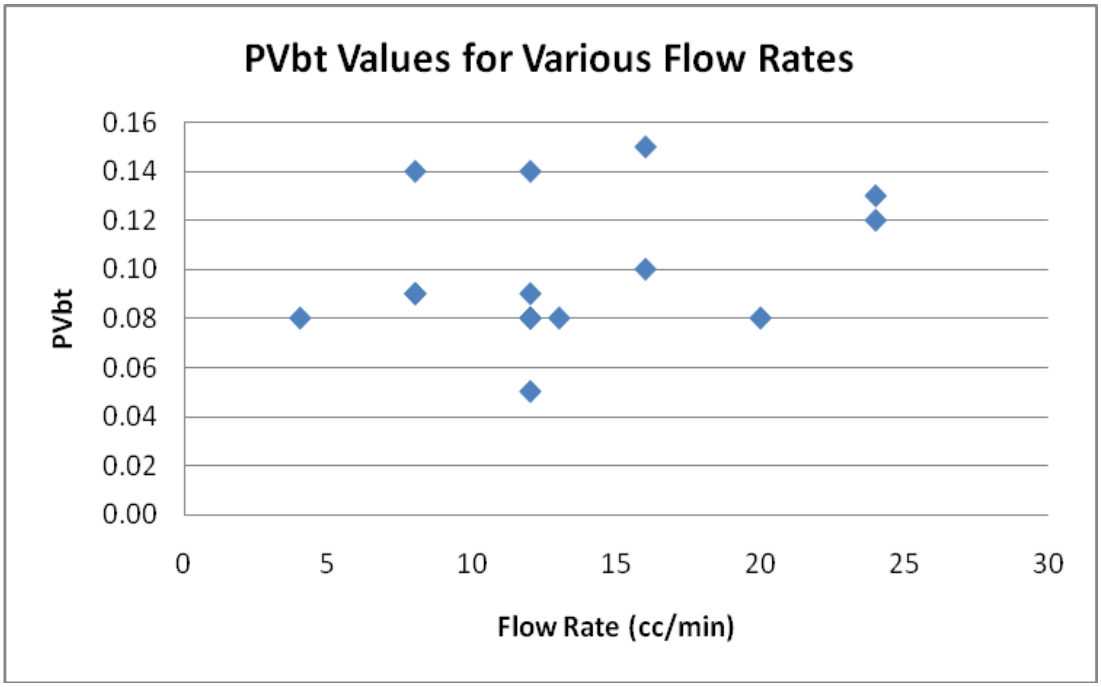

Fig 4.2. Pore Volumes to Breakthrough at Various Flow Rates.

For these vuggy core samples, the PVbt values are significantly lower than those in previous research for carbonates.

The process discussed in Chapter II was used for every core sample to obtain the data described above. Individual core sample plots, data, and figures can be viewed in the appendices.

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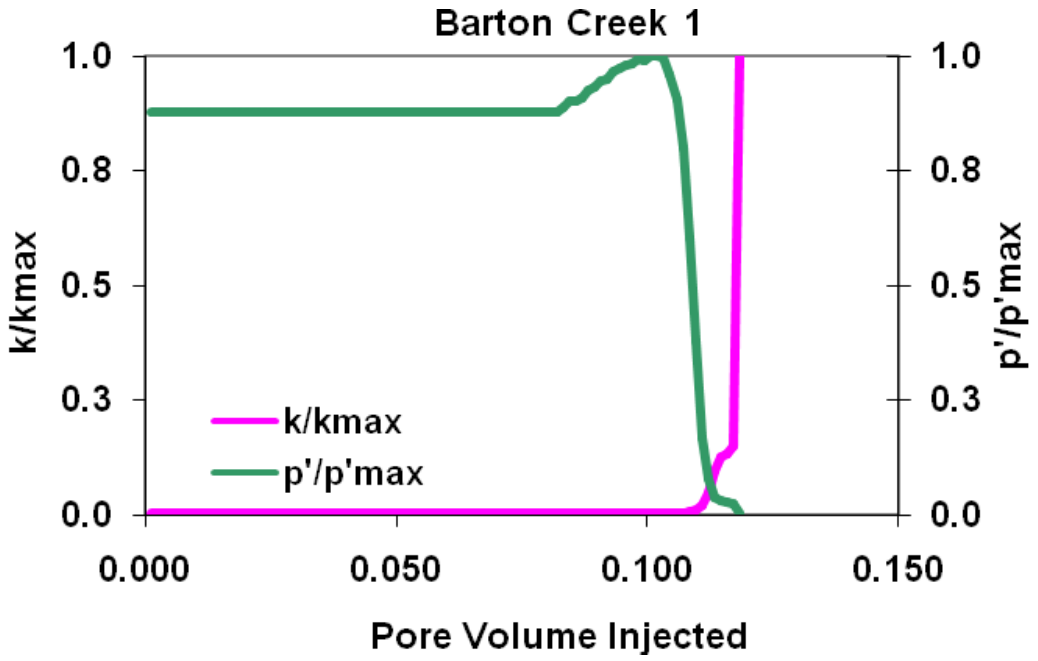
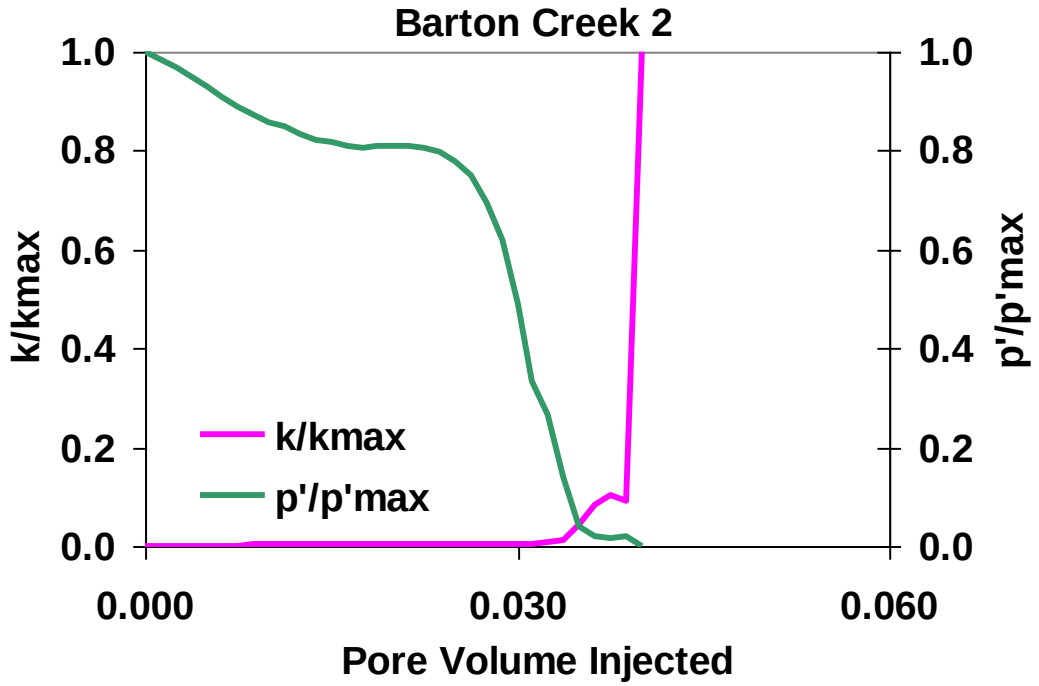
APPENDIX A

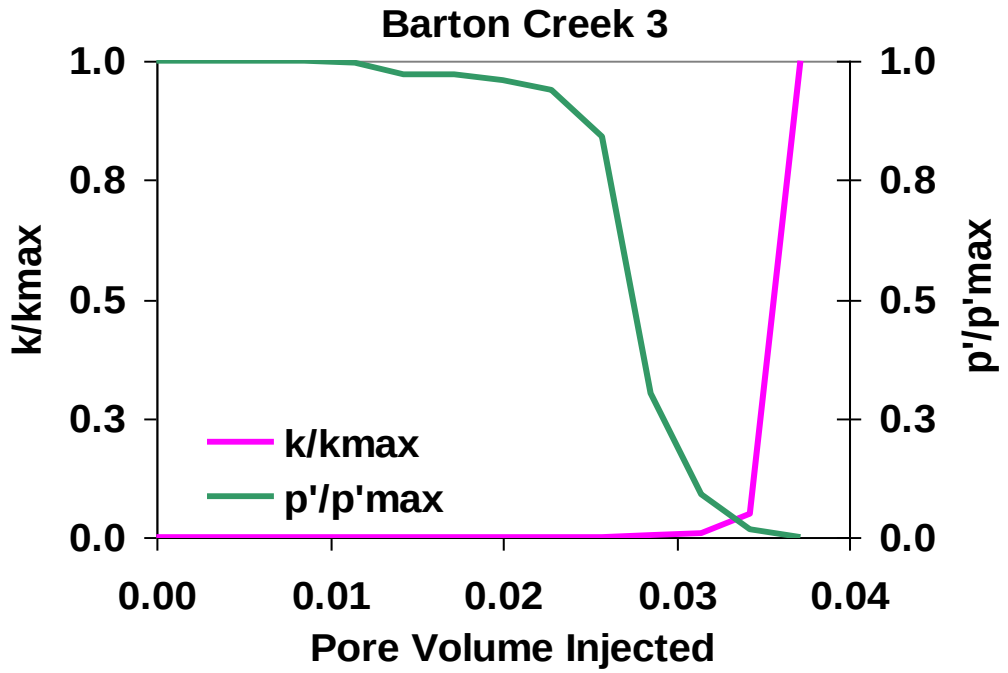
NORMALIZED PRESSURE AND PERMEABILITY PLOTS FOR BARTON CREEK SAMPLES



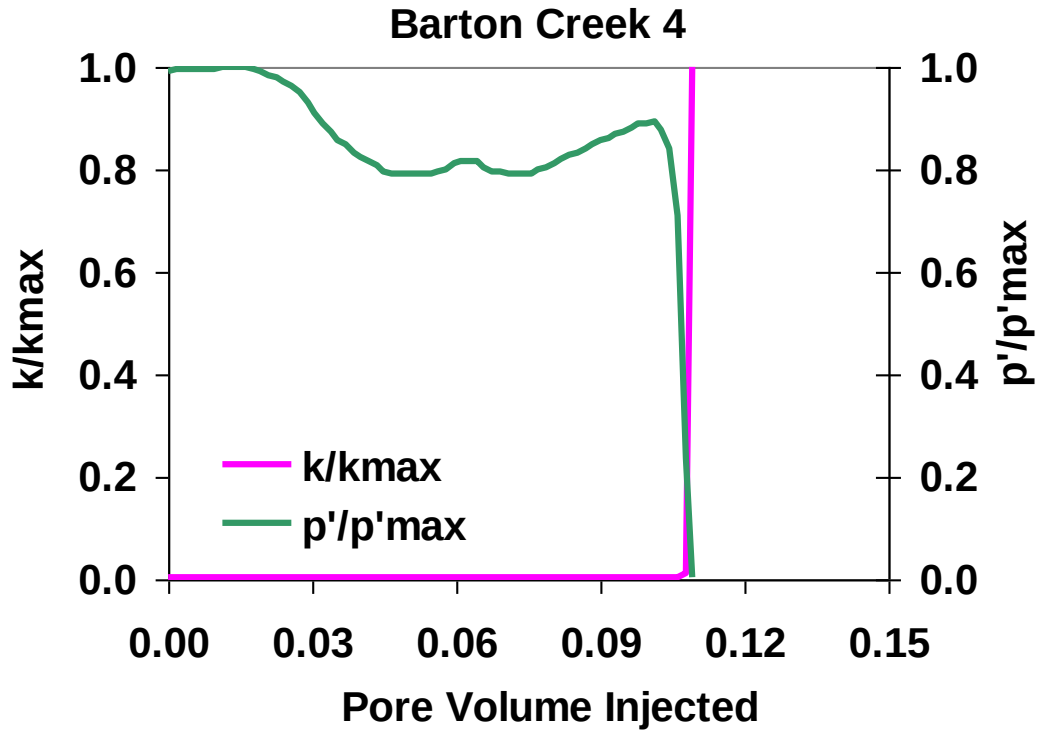
BC1 Pressure and Permeability as a Function of Pore Volumes Injected



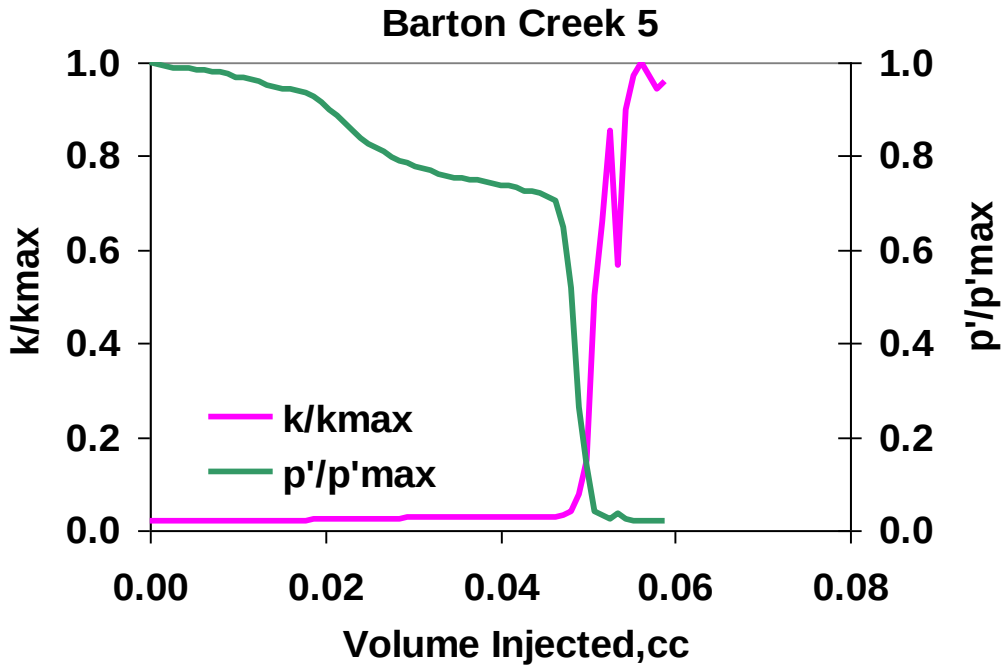
BC2 Pressure and Permeability as a Function of Pore Volumes Injected



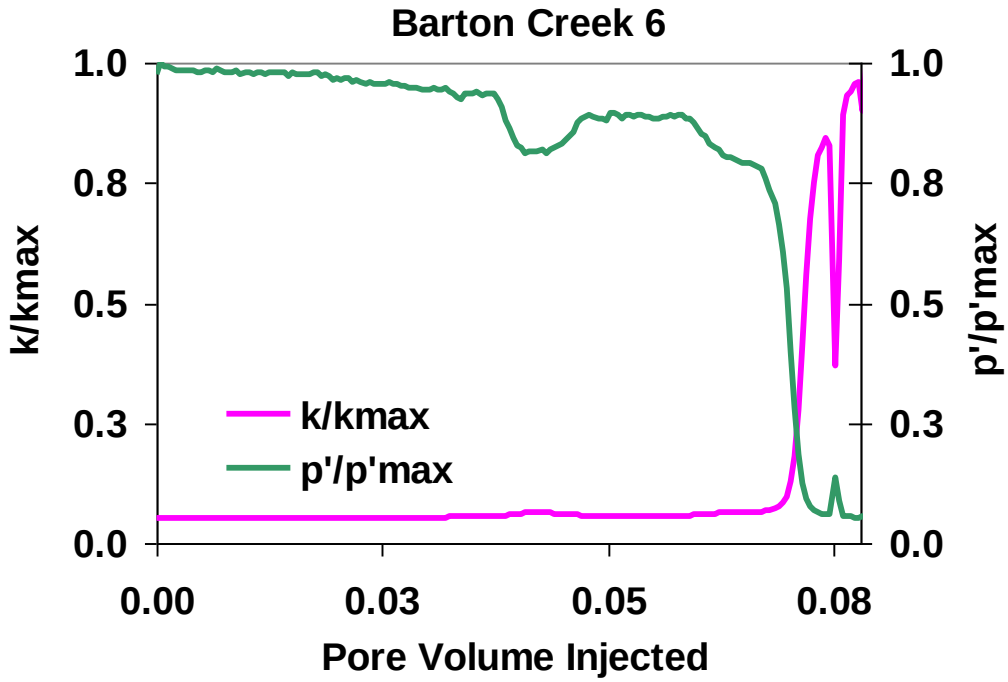
BC3 Pressure and Permeability as a Function of Pore Volumes Injected



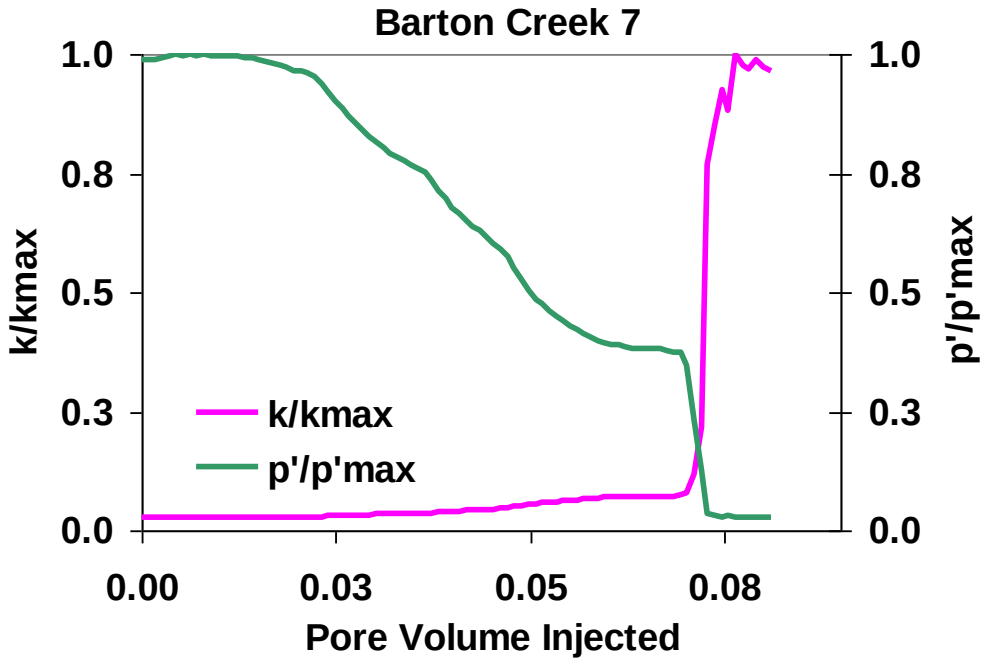
BC4 Pressure and Permeability as a Function of Pore Volumes Injected



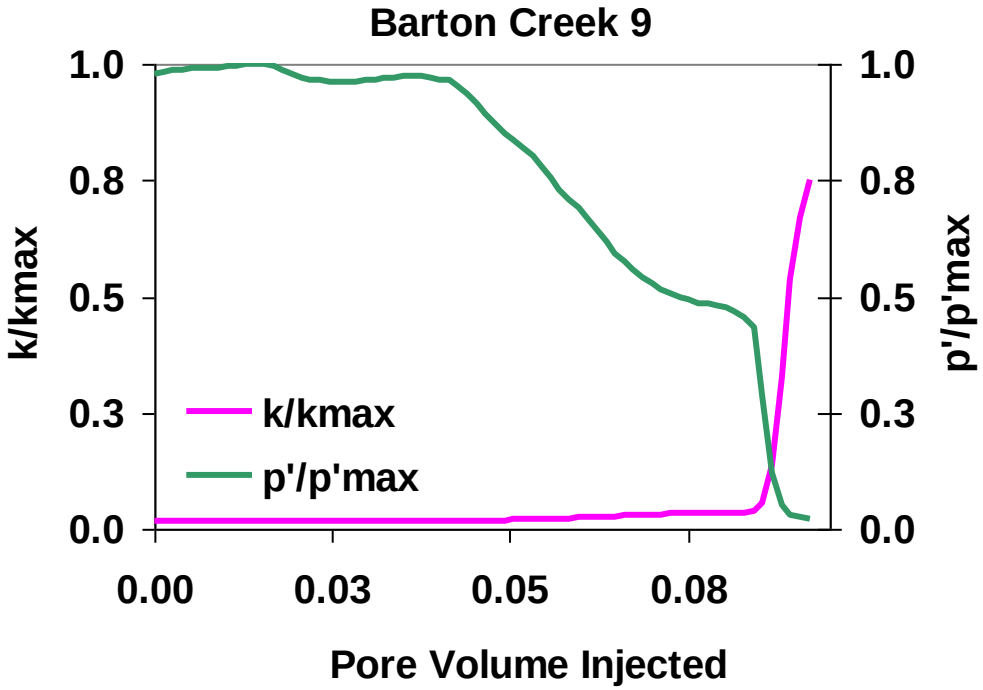
BC5 Pressure and Permeability as a Function of Pore Volumes Injected



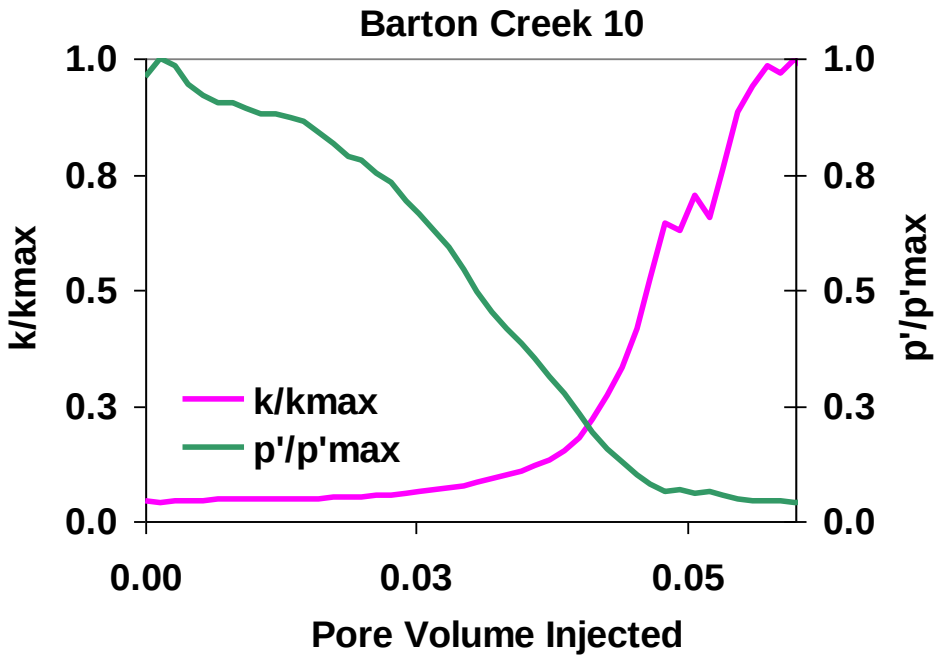
BC6 Pressure and Permeability as a Function of Pore Volumes Injected



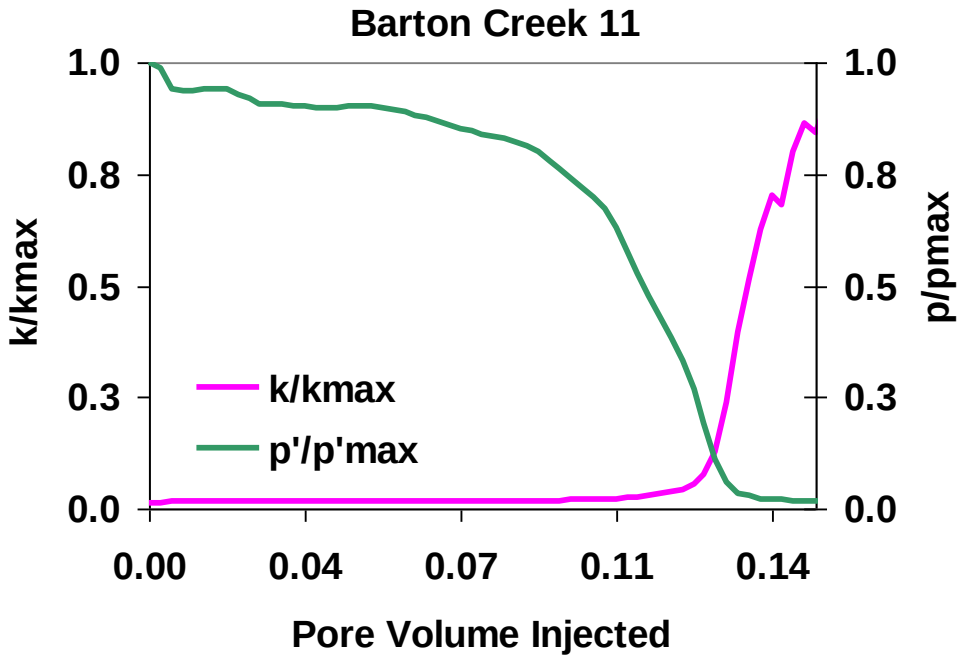
BC7 Pressure and Permeability as a Function of Pore Volumes Injected



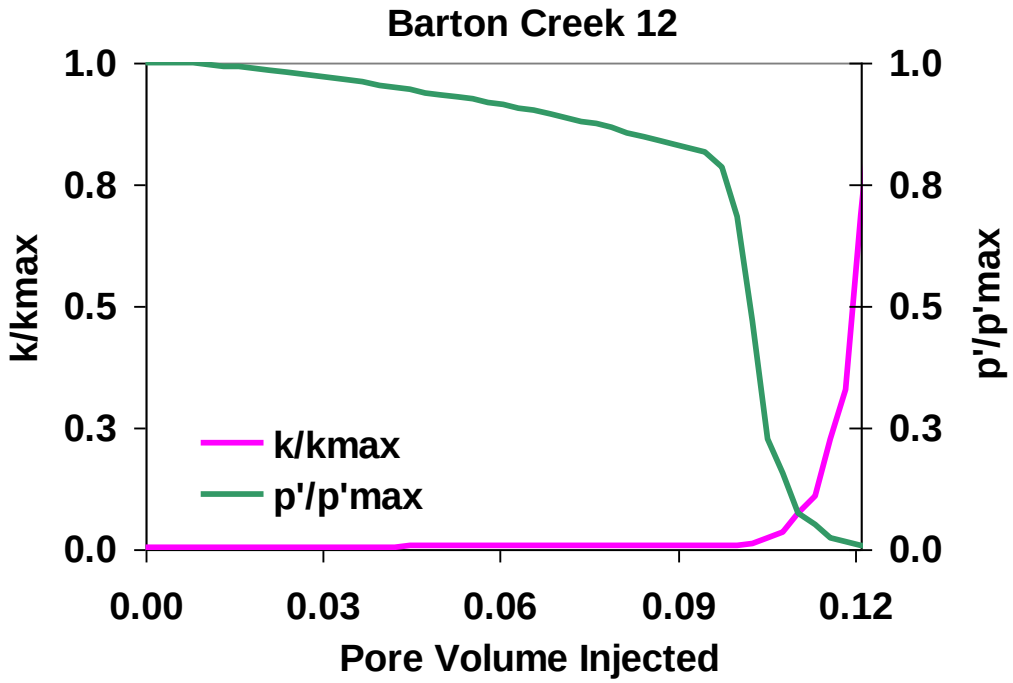
BC9 Pressure and Permeability as a Function of Pore Volumes Injected



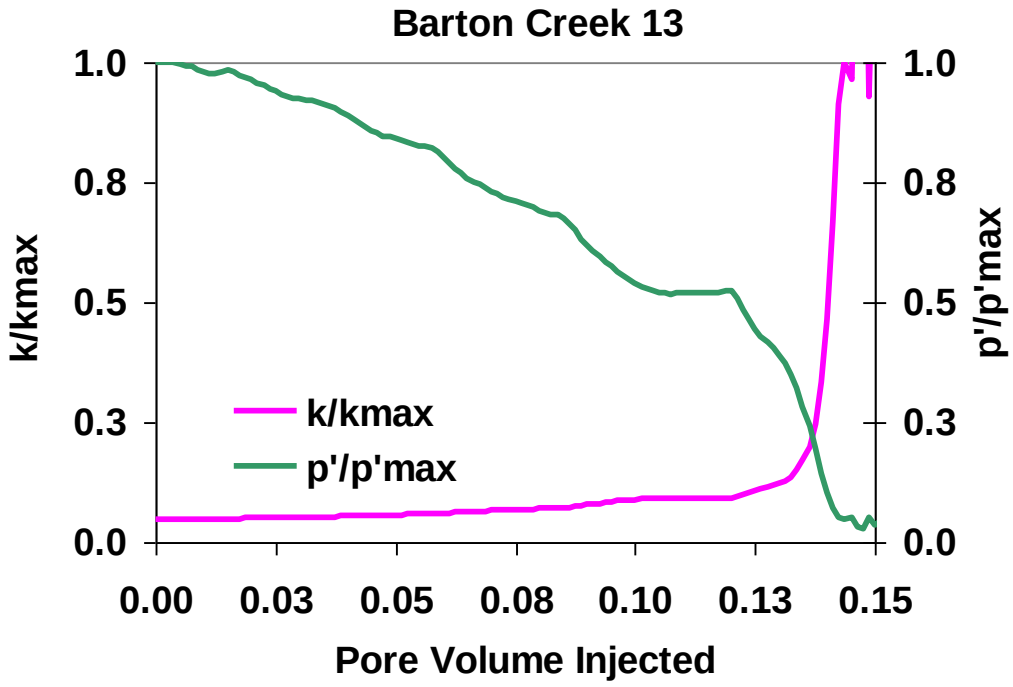
BC10 Pressure and Permeability as a Function of Pore Volumes Injected



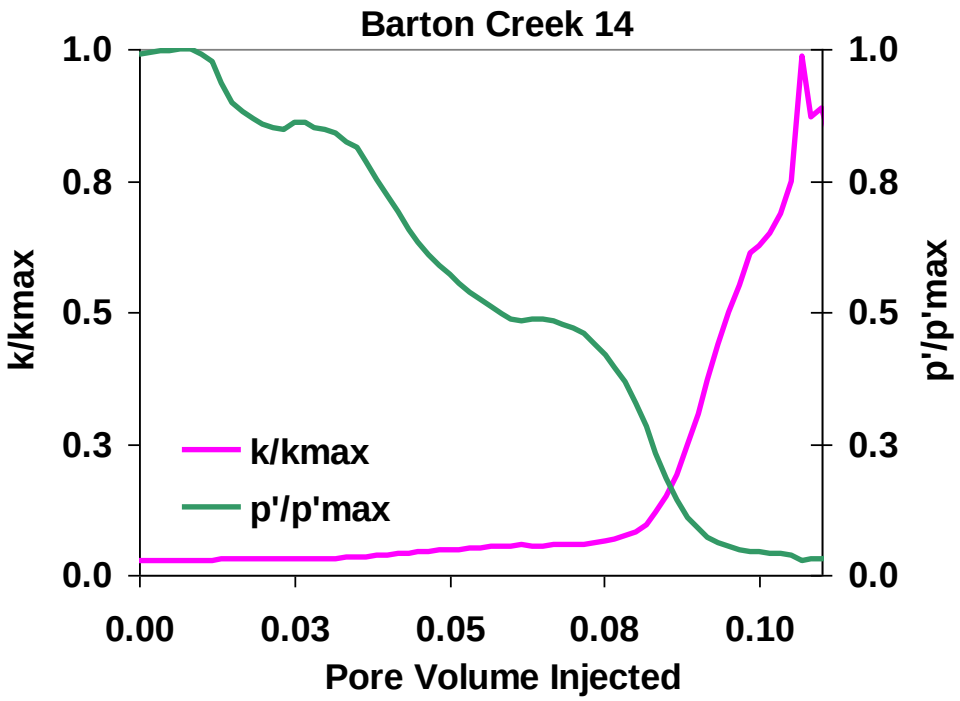
BC11 Pressure and Permeability as a Function of Pore Volumes Injected



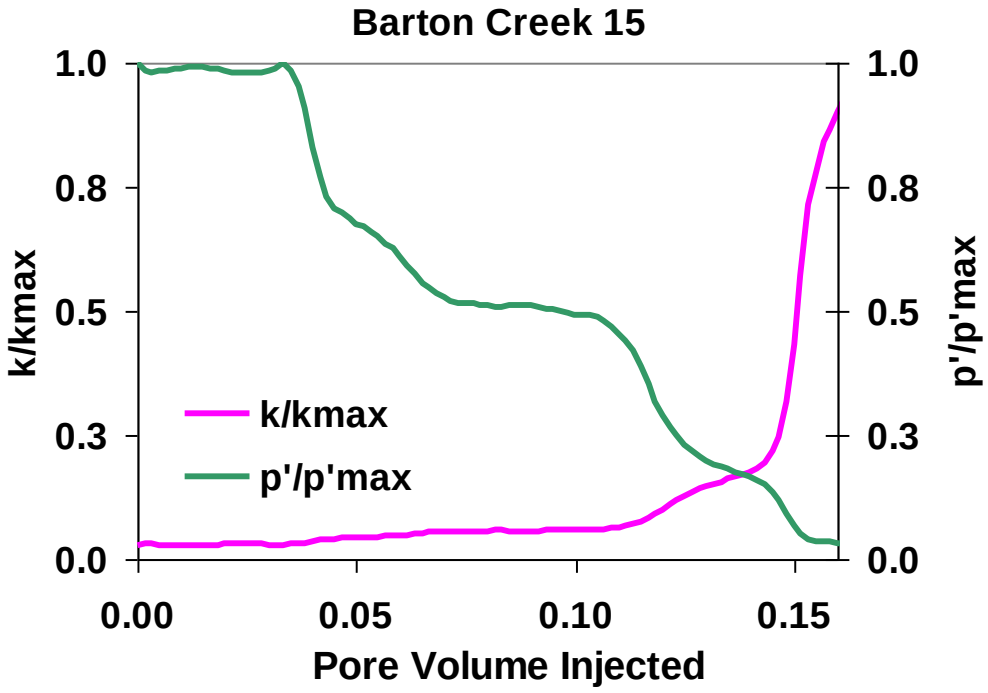
BC12 Pressure and Permeability as a Function of Pore Volumes Injected



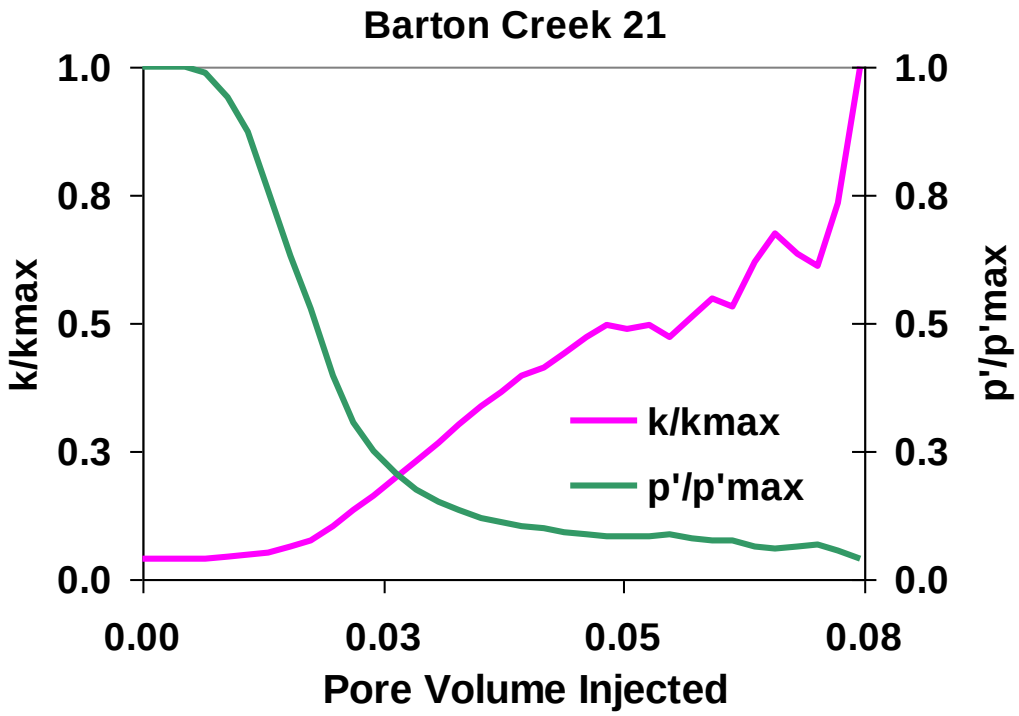
BC13 Pressure and Permeability as a Function of Pore Volumes Injected



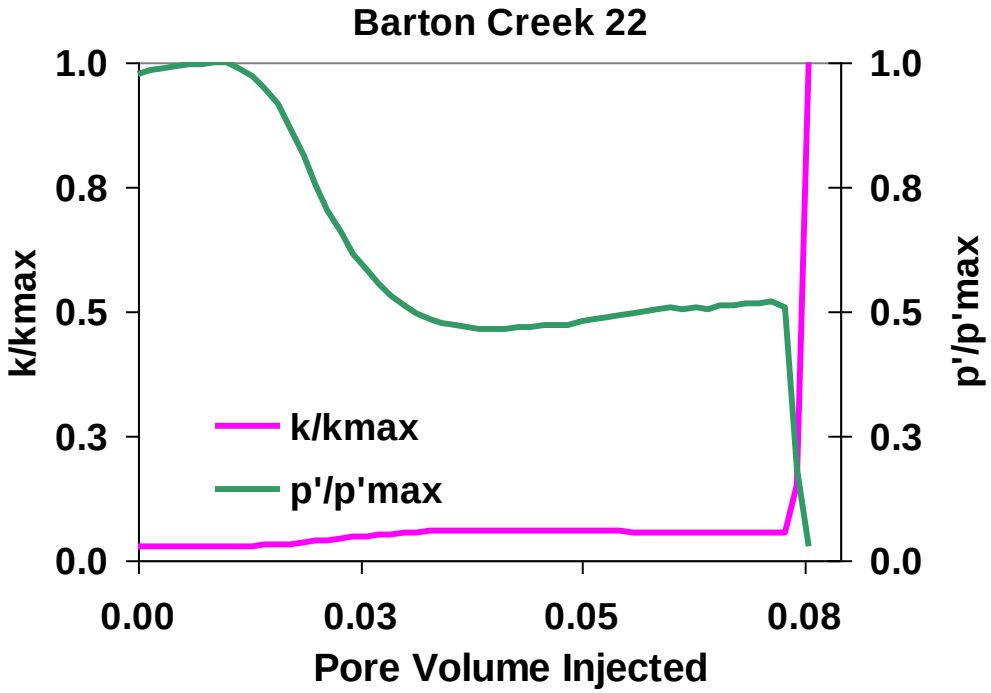
BC14 Pressure and Permeability as a Function of Pore Volumes Injected



BC15 Pressure and Permeability as a Function of Pore Volumes Injected



BC21 Pressure and Permeability as a Function of Pore Volumes Injected



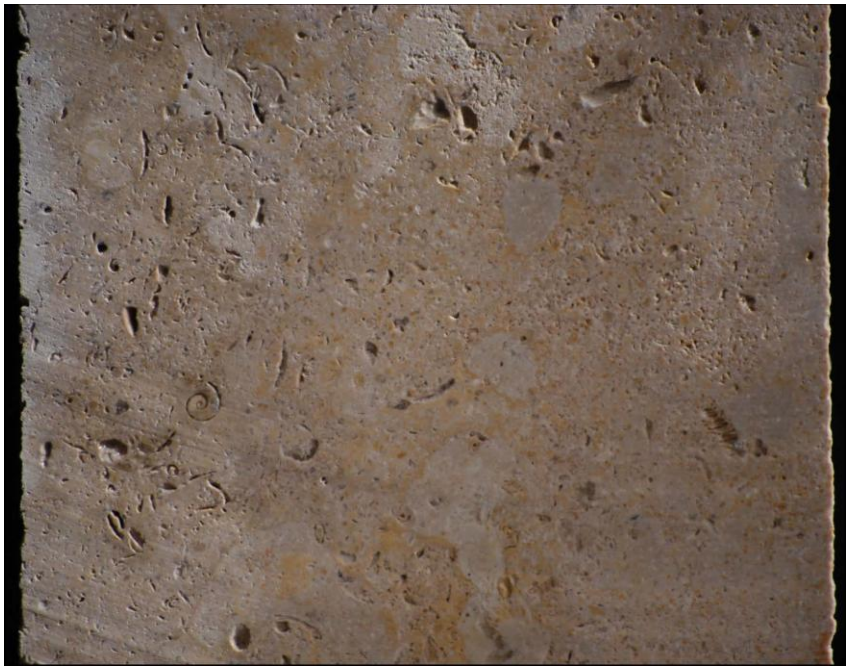
BC22 Pressure and Permeability as a Function of Pore Volumes Injected

APPENDIX B

BARTON CREEK SAMPLE CLASSIFICATION

Geological Classifications for the Barton Creek samples were done by Reine Vera under the direction of Dr. Wayne Ahr. A more thorough discussion is included in her thesis.

Sample #	Barton Creek-0
Date Received	11/13/2007
Location	AUSTIN, TX (Southwest Parkway & Highway 360)
Formation	EDWARD LIMESTONE

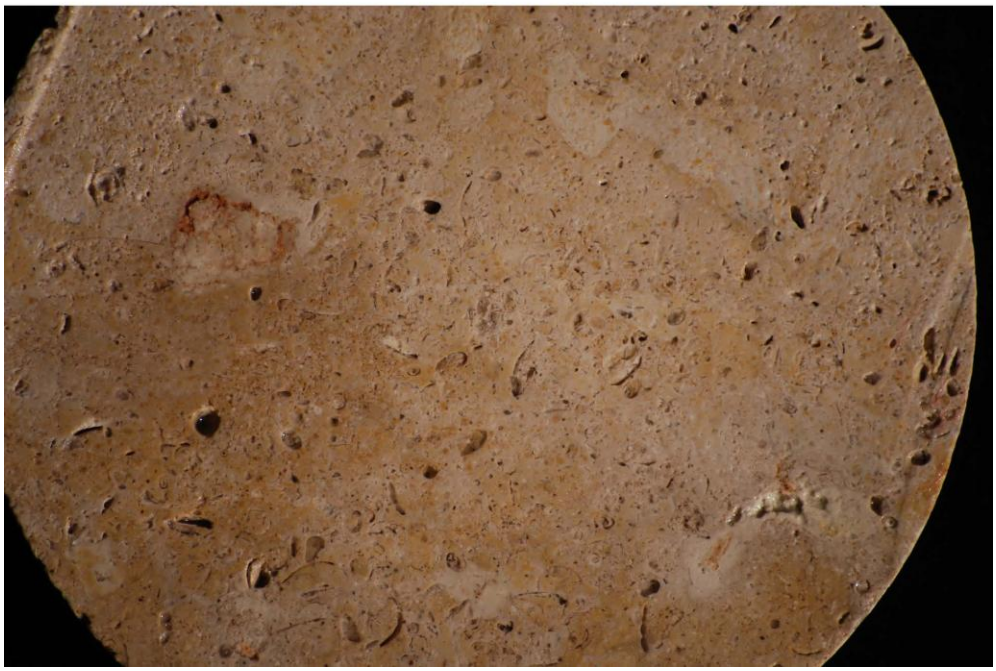


Rock Type	LIMESTONE
Color	Yellowish white
Bedding & Sedimentary Structures	Burrows filled with lighter color, finer grain (upperlower fine) of skeletal debris, pelloids, and mud.
Dunham Classification	PACKSTONE / GRAINSTONE mixture (Skeletal debris Packstone / Grainstone with some pelloids).
Main Grain Types	Skeletal debris (Foraminifers benthonic & planktonic, abundant Miliolid, Gastropods, bivalves fossil shells) with the walls condition dissolved and filled by calcite in many places. Pelloids are present in some places.
Visible Porosity	Abundant pelloid and skeletal moldics, interparticle,intraparticle (in some places due to the solution of internal sediment filling of fossils), dissolution enhanced, vuggy (highly dissolved skeletal moldics were becoming vug in some places).
Cement/Matrix	Calcite as cement and matrix
Accessory Minerals	
Oil Stain	No
Genetic Classification of Carbonate Porosity (Ahr)	Hybrid 1 (hybrid of depositional and diagenetic processes)

Sample #	Barton Creek-1
Date Received	1/18/2008
Location	AUSTIN, TX (Southwest Parkway & Highway 360)
Formation	EDWARD LIMESTONE

Sample #	Barton Creek-1
Date Received	1/18/2008
Location	AUSTIN, TX (Southwest Parkway & Highway 360)
Formation	EDWARD LIMESTONE
Rock Type	LIMESTONE
Color	Yellowish white
Bedding & Sedimentary Structures	Burrows filled with lighter color, finer grain (upperlower fine) of skeletal debris, pelloids, and mud.
Dunham Classification	PACKSTONE / GRAINSTONE mixture (Skeletal debris Packstone / Grainstone with some pelloids).
Main Grain Types	Skeletal debris (Foraminifers benthonic & planktonic, abundant Miliolid, Gastropods, bivalves fossil shells) with the walls condition dissolved and filled by calcite in many places. Pelloids are present in some places.
Visible Porosity	Abundant pelloid and skeletal moldics, interparticle,intraparticle (in some places due to the solution of internal sediment filling of fossils), dissolution enhanced, vuggy (highly dissolved skeletal moldics were becoming vug in some places).
Cement/Matrix	Calcite as cement and matrix
Accessory Minerals	
Oil Stain	No
Genetic Classification of Carbonate Porosity (Ahr)	Hybrid 1 (hybrid of depositional and diagenetic processes)

Sample #	Barton Creek-2
Date Received	1/18/2008
Location	AUSTIN, TX (Southwest Parkway & Highway 360)
Formation	EDWARD LIMESTONE



Rock Type	LIMESTONE
Color	Yellowish white
Bedding & Sedimentary Structures	Bedding-like structure where the sample consists of two different parts –the lighter color has more mud and the other has more grains. Burrows filled with lighter color, finer grain (upper-lower fine) of skeletal debris, pelloids, and mud.
Dunham Classification	PACKSTONE / GRAINSTONE mixture (Skeletal debris or bioclastic Packstone / Grainstone with some pelloids).
Main Grain Types	Skeletal debris (Foraminifers benthonic & planktonic, Miliolid, Fusulinid, Lepidocyclina sp.?, and bivalves fossil shell) with the walls condition dissolved and filled by calcite. Pelloids are present in some places. Intraclast consist of finer grain, fossil debris and oxidized (red) minerals. Dendritic growth forms of the widespread calcimicrobe organism (Epiphyton) are present in some places.
Visible Porosity	Abundant pelloid and skeletal moldics, interparticle,intraparticle (in some places due to the solution of internal sediment filling of fossils), dissolution enhanced, vuggy (highly dissolved skeletal moldics were becoming vug in some places).
Cement/Matrix	Calcite as cement and matrix
Accessory Minerals	
Oil Stain	No
Genetic Classification of Carbonate Porosity (Ahr)	Hybrid 1 (hybrid of depositional and diagenetic processes)

Sample #	Barton Creek-3
Date Received	1/18/2008
Location	AUSTIN, TX (Southwest Parkway & Highway 360)
Formation	EDWARD LIMESTONE

Rock Type	LIMESTONE
Color	Yellowish white
Bedding & Sedimentary Structures	Burrows filled with lighter color, finer grain (upper: lower fine) of skeletal debris, pelloids, and mud.
Dunham Classification	PACKSTONE / GRAINSTONE mixture (Skeletal debris or bioclastic Packstone / Grainstone with some pelloids).
Main Grain Types	Skeletal debris (Foraminifers benthonic & planktonic, Miliolid, Fusulinid, Lepidocyclina sp., and bivalves fossil shell) with the walls condition dissolved and filled by calcite in many places. Pelloids are present in some places. Intraclast consist of finer grain, fossil debris and calcite crystal inclusion. Red (oxidized) minerals are present in some places.
Visible Porosity	Abundant pelloid and skeletal moldics, interparticle,intraparticle (in some places due to the solution of internal sediment filling of fossils), dissolution enhanced, vuggy (highly dissolved skeletal moldics were becoming vug in some places).
Cement/Matrix	Calcite as cement and matrix
Accessory Minerals	
Oil Stain	No
Genetic Classification of Carbonate Porosity (Ahr)	Hybrid 1 (hybrid of depositional and diagenetic processes)

Sample #	Barton Creek-4
Date Received	1/18/2008
Location	AUSTIN, TX (Southwest Parkway & Highway 360)
Formation	EDWARD LIMESTONE

Rock Type	LIMESTONE
Color	Yellowish white
Bedding & Sedimentary Structures	Burrows filled with lighter color, finer grain (upperlower fine) of skeletal debris, pelloids, mud, and calcite crystals.
Dunham Classification	PACKSTONE / GRAINSTONE mixture (Skeletal debris or bioclastic Packstone / Grainstone with some pelloids).
Main Grain Types	Skeletal debris (Foraminifers benthonic & planktonic, Miliolid, Fusulinid, Gastropods and bivalves fossil shell) with the walls condition dissolved and filled by calcite. Pelloids are present in some places. Intraclast consist of finer grain, fossil debris and calcite crystals.
Visible Porosity	Abundant pelloid and skeletal moldics, interparticle,intraparticle (in some places due to the solution of internal sediment filling of fossils), dissolution enhanced, vuggy (highly dissolved skeletal moldics were becoming vug in some places).
Cement/Matrix	Calcite as cement and matrix
Accessory Minerals	
Oil Stain	No
Genetic Classification of Carbonate Porosity (Ahr)	Hybrid 1 (hybrid of depositional and diagenetic processes)

Sample #	Barton Creek-7
Date Received	1/18/2008
Location	AUSTIN, TX (Southwest Parkway & Highway 360)
Formation	EDWARD LIMESTONE

Rock Type	LIMESTONE
Color	Yellowish white
Bedding & Sedimentary Structures	Burrows filled with lighter color, finer grain (upperlower fine) of skeletal debris, and mud. Some have been oxidized.
Dunham Classification	PACKSTONE / GRAINSTONE mixture (Skeletal debris or bioclastic Packstone / Grainstone with some pelloids).
Main Grain Types	Skeletal debris (Foraminifers benthonic & planktonic, Miliolid, and bivalves fossil shells) with the walls condition dissolved and filled by calcite in many places. Pelloids are present in some places. Intraclast consist of finer grain and fossil debris. Dendritic growth forms of the widespread calcimicrobe organism (Epiphyton) are present in some places.
Visible Porosity	Abundant pelloid and skeletal moldics, interparticle,intraparticle (in some places due to the solution of internal sediment filling of fossils), dissolution enhanced, vuggy (highly dissolved skeletal moldics were becoming vug in some places).
Cement/Matrix	Calcite as cement and matrix
Accessory Minerals	
Oil Stain	No
Genetic Classification of Carbonate Porosity (Ahr)	Hybrid 1 (hybrid of depositional and diagenetic processes)

Sample #	Barton Creek-20
Date Received	5/2/2008
Location	AUSTIN, TX (Southwest Parkway & Highway 360)
Formation	EDWARD LIMESTONE

Rock Type	LIMESTONE
Color	White
Bedding & Sedimentary Structures	Slightly burrowed, filled of skeletal debris, peloids, and mud.
Dunham Classification	GRAINSTONE/PACKSTONE mixture, with some peloids (oolid?).
Main Grain Types	Skeletal debris (Miliolid and benthic foraminifers) with the walls condition dissolved and filled by calcite in many places.
Visible Porosity	Intergranular, some porous some cemented, skeletal moldics, slightly vuggy
Cement/Matrix	Calcite as cement and matrix
Accessory Minerals	Calcite
Oil Stain	No
Genetic Classification of Carbonate Porosity (Ahr)	Hybrid 1 (hybrid of depositional and diagenetic processes)

Sample #	Barton Creek-21
Date Received	5/2/2008
Location	AUSTIN, TX (Southwest Parkway & Highway 360)
Formation	EDWARD LIMESTONE

Rock Type	LIMESTONE
Color	Brownish White
Bedding & Sedimentary Structures	Slightly burrowed, filled of skeletal debris, peloids, and mud.
Dunham Classification	GRAINSTONE/PACKSTONE mixture, with some peloids (oolid?).
Main Grain Types	Skeletal debris (Miliolid and biserial benthic foraminifers, Moluscs) with the walls condition dissolved and filled by calcite in many places, oolid (?).
Visible Porosity	Abundant skeletal moldic, interparticle, vuggy
Cement/Matrix	Calcite as cement and matrix
Accessory Minerals	Calcite
Oil Stain	No
Genetic Classification of Carbonate Porosity (Ahr)	Hybrid 1 (hybrid of depositional and diagenetic processes)

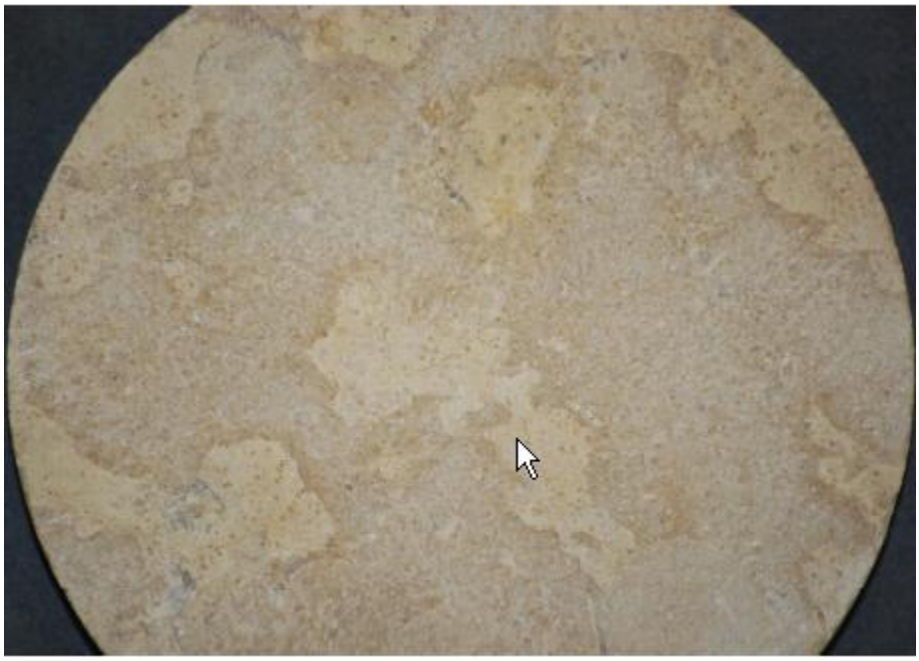
Sample #	Barton Creek-22
Date Received	5/2/2008
Location	AUSTIN, TX (Southwest Parkway & Highway 360)
Formation	EDWARD LIMESTONE

Rock Type	LIMESTONE
Color	White
Bedding & Sedimentary Structures	
Dunham Classification	GRAINSTONE/PACKSTONE mixture, with some peloids (oolid?).
Main Grain Types	Skeletal debris (Miliolid and benthic foraminifers) with the walls condition dissolved and filled by calcite in many places.
Visible Porosity	Intergranular, some porous some cemented, skeletal moldics, slightly vuggy
Cement/Matrix	Calcite as cement and matrix
Accessory Minerals	Calcite
Oil Stain	No
Genetic Classification of Carbonate Porosity (Ahr)	Hybrid 1 (hybrid of depositional and diagenetic processes)

Sample #	Barton Creek-23
Date Received	5/2/2008
Location	AUSTIN, TX (Southwest Parkway & Highway 360)
Formation	EDWARD LIMESTONE

Rock Type	LIMESTONE
Color	White
Bedding & Sedimentary Structures	
Dunham Classification	GRAINSTONE/PACKSTONE mixture, with some peloids (oolid?).
Main Grain Types	Skeletal debris (Gastropods and benthic foraminifers) with the walls condition dissolved and filled by calcite in many places.
Visible Porosity	Intergranular, a lot of vuggy porosity, skeletal moldics, very good porosity.
Cement/Matrix	Calcite as cement and matrix
Accessory Minerals	Calcite
Oil Stain	No
Genetic Classification of Carbonate Porosity (Ahr)	Hybrid 1 (hybrid of depositional and diagenetic processes)

Sample #	Barton Creek-24
Date Received	5/2/2008
Location	AUSTIN, TX (Southwest Parkway & Highway 360)
Formation	EDWARD LIMESTONE



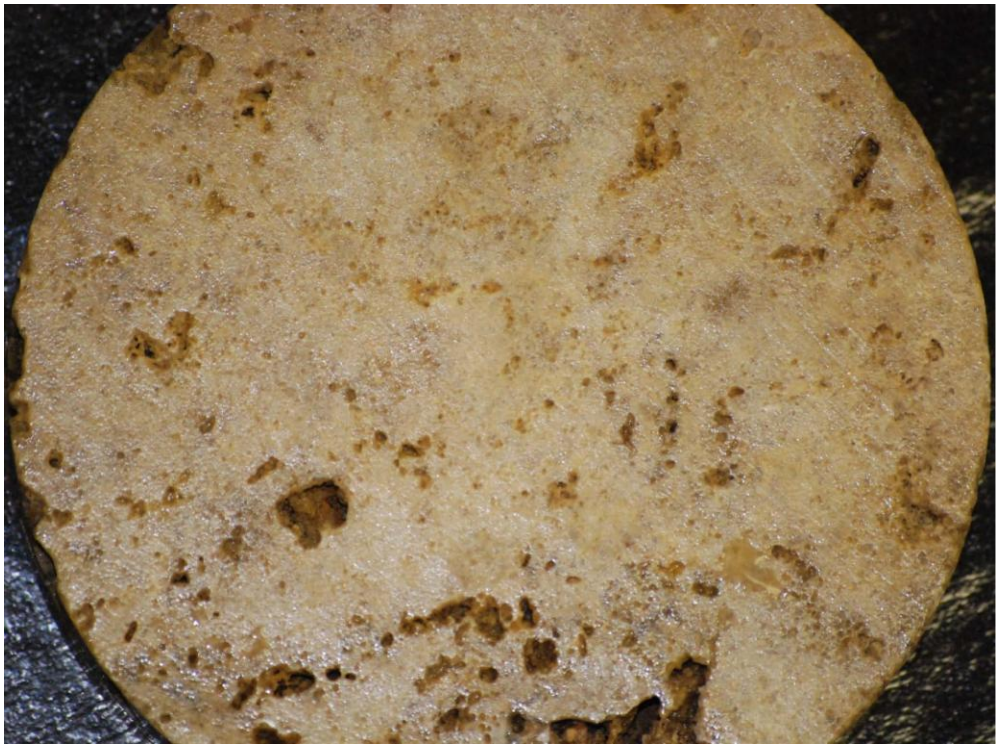
Rock Type	LIMESTONE
Color	Brownish White
Bedding & Sedimentary Structures	Highly burrowed, filled lighter color, finer grain, skeletal debris, peloids, and mud.
Dunham Classification	PACKSTONE/ WACKESTONE(in the burrow) mixture, with some peloids (oolid?).
Main Grain Types	Skeletal debris (Miliolid and biserial benthic foraminifers, Moluscs, bivalve fragments) with the walls condition dissolved and filled by calcite in many places, oolid (?).
Visible Porosity	A lot of mud lowering porosity, micro porosity, skeletal moldic in some places.
Cement/Matrix	Calcite as cement and matrix
Accessory Minerals	Calcite
Oil Stain	No
Genetic Classification of Carbonate Porosity (Ahr)	Hybrid 1 (hybrid of depositional and diagenetic processes)

APPENDIX C

CHURCH ROAD SAMPLE CLASSIFICATION

Geological Classifications for the Church Road samples were done by Reine Vera under the direction of Dr. Wayne Ahr. A more thorough discussion is included in her thesis.

Sample #	CR-1
Date Received	3/20/2008
Location	College Station, TX
Formation	Unknown

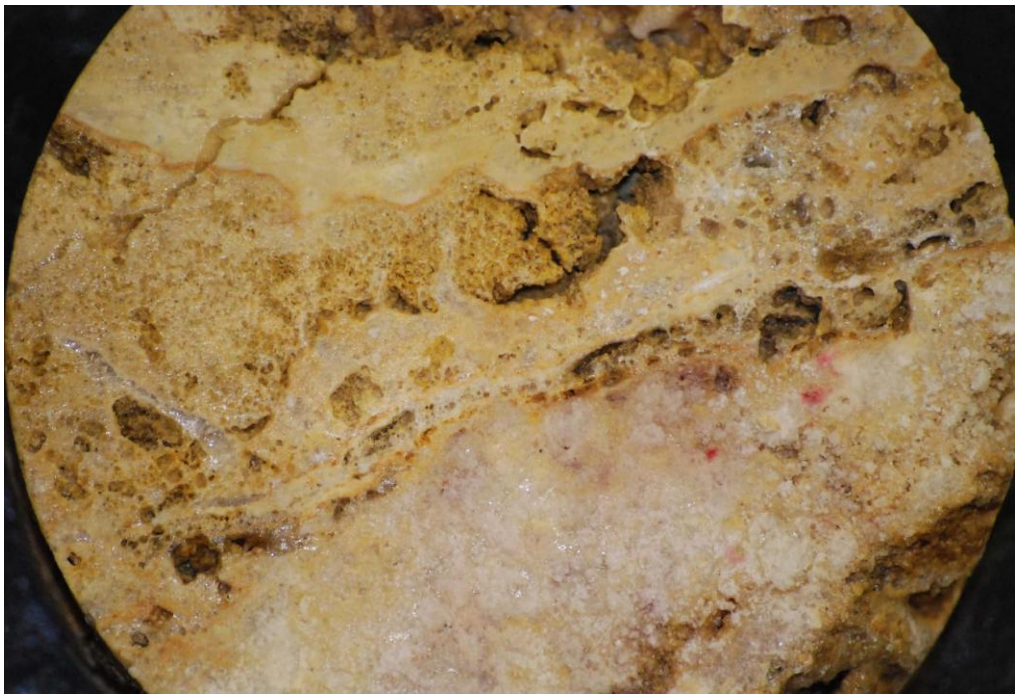


Rock Type	DOLOMITIC LIMESTONE
Color	Yellowish grey
Bedding & Sedimentary Structures	No visible original depositional texture. Diagenetic altered and completely recrystallized.
Dunham Classification	DOLOMITIZED PELOIDAL GRAINSTONE (The original rock is a peloidal grainstone, then altered through diagenetic process, replaced extensively by dolomite)
Main Grain Types	Non skeletal/ peloid (oolite?), and minor skeletal fragments
Visible Porosity	Intercrystalline, vuggy (strongly leached), and moldic (replacement of precursor limestone followed by probable dissolution of undolomitized limestone remnants)
Cement/Matrix	Calcite as cement and matrix
Accessory Minerals	Dolomite
Oil Stain	No
Genetic Classification of Carbonate Porosity (Ahr)	Hybrid 1 (hybrid of depositional and diagenetic processes)

Sample #	CR-2
Date Received	3/20/2008
Location	College Station, TX
Formation	Unknown

Rock Type	DOLOMITIC LIMESTONE
Color	Yellowish grey
Bedding & Sedimentary Structures	No visible original depositional texture. Diagenetic altered and completely recrystallized. (diagenetic process gives structure of coarsely crystalline calcite filled pore spaces).
Dunham Classification	DOLOMITIZED PELOIDAL GRAINSTONE (The original rock is a peloidal grainstone, then altered through diagenetic process, replaced extensively by dolomite)
Main Grain Types	Non skeletal/ peloid and crystalline
Visible Porosity	Intercrystalline, vug (strongly leached) and moldic
Cement/Matrix	Calcite as cement and matrix
Accessory Minerals	Dolomite
Oil Stain	No
Genetic Classification of Carbonate Porosity (Ahr)	Hybrid 1 (hybrid of depositional and diagenetic processes)

Sample #	CR-3
Date Received	3/20/2008
Location	College Station, TX
Formation	Unknown

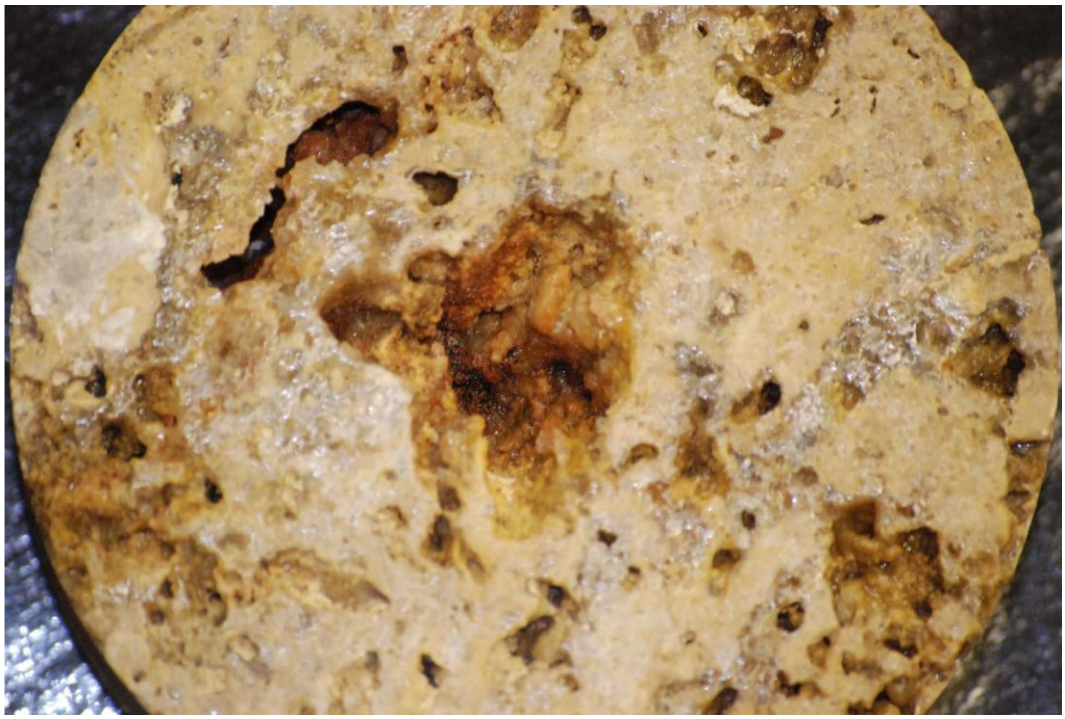


Rock Type	DOLOMITIC LIMESTONE
Color	White and yellow layers
Bedding & Sedimentary Structures	Even bedding, it's divided into two sections: one section is dolomitized and the other is extensively leached. Structure after diagenetic process: solution seams.
Dunham Classification	DOLOMITIZED PELOIDAL GRAINSTONE (The original rock is a peloidal grainstone then altered by diagenetic process, replaced extensively by dolomite)
Main Grain Types	Non skeletal/ peloid (oolite?) and crystalline
Visible Porosity	Intercrystalline, vuggy (strongly leached), peloidal moldic, and fenestral fabric. Most of porosity was occluded by a coarse meshwork of calcite crystal laths.
Cement/Matrix	Calcite as cement and matrix
Accessory Minerals	Dolomite and silica mineral (probably Chalcedony)
Oil Stain	No
Genetic Classification of Carbonate Porosity (Ahr)	Hybrid 1 (hybrid of depositional and diagenetic processes)

Sample #	CR-4
Date Received	3/20/2008
Location	College Station, TX
Formation	Unknown

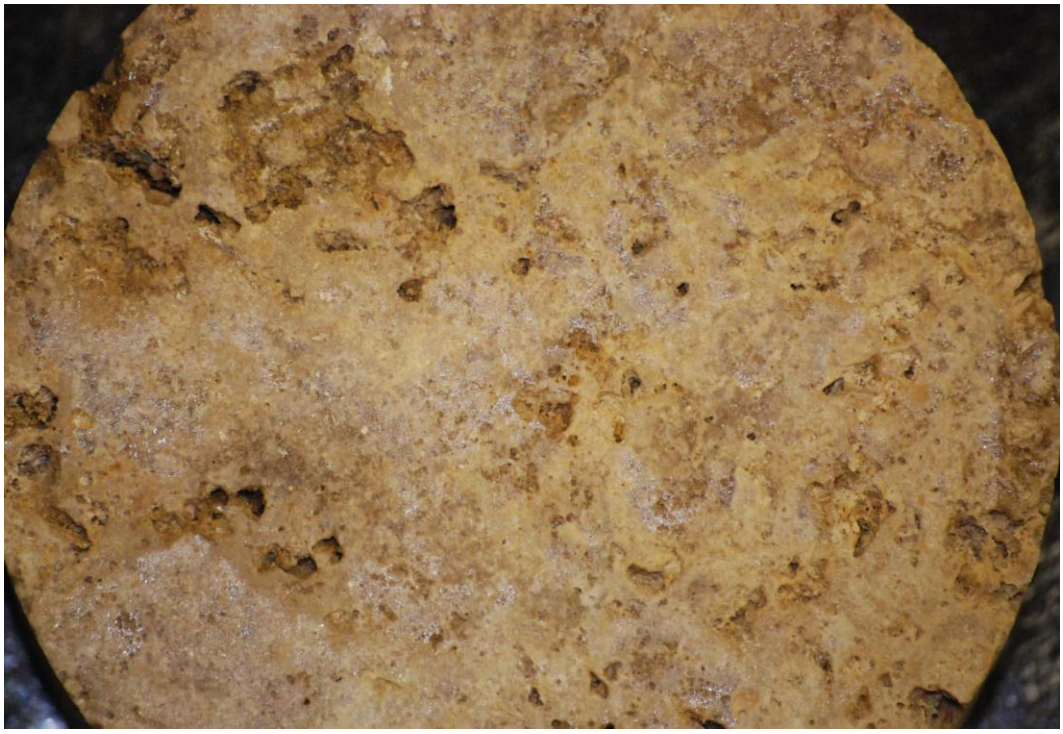
Rock Type	DOLOMITIC LIMESTONE
Color	Yellowish grey
Bedding & Sedimentary Structures	No visible original depositional texture. Diagenetic altered and completely recrystallized.
Dunham Classification	DOLOMITIZED PELOIDAL GRAINSTONE (The original rock is a peloidal grainstone, then altered through diagenetic process, replaced extensively by dolomite)
Main Grain Types	Non skeletal/ peloid and crystalline
Visible Porosity	Intercrystalline, vug (strongly leached) or cavern in some places, and peloidal moldic
Cement/Matrix	Calcite as cement and matrix
Accessory Minerals	Dolomite
Oil Stain	No
Genetic Classification of Carbonate Porosity (Ahr)	Hybrid 1 (hybrid of depositional and diagenetic processes)

Sample #	CR-5
Date Received	3/20/2008
Location	College Station, TX
Formation	Unknown



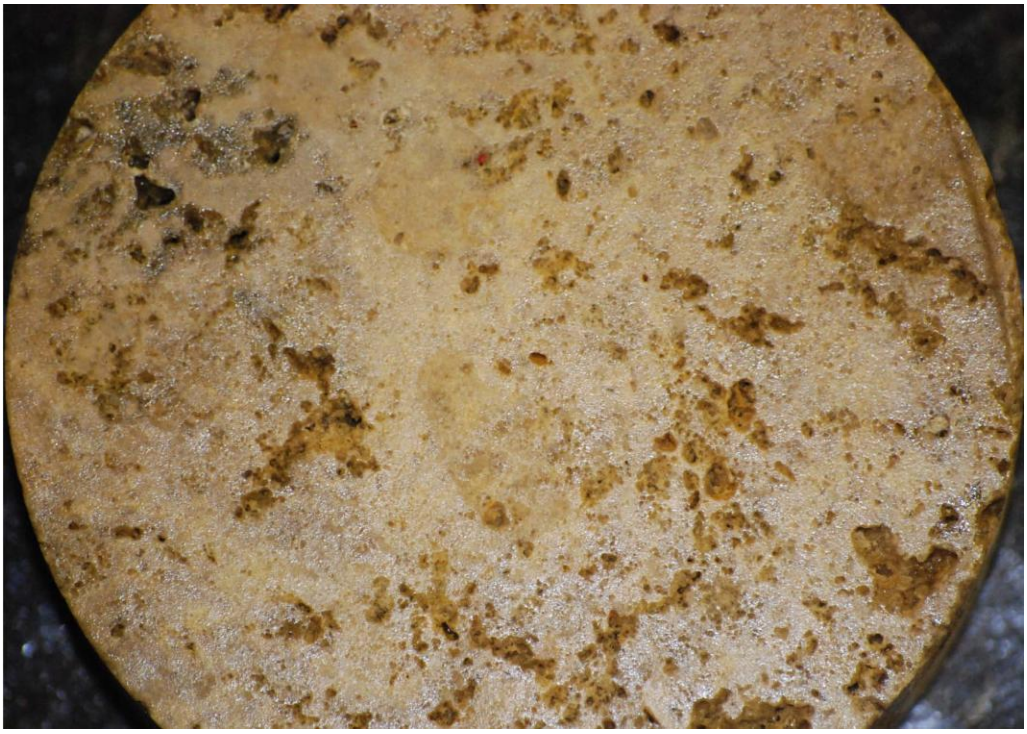
Rock Type	DOLOMITIC LIMESTONE
Color	Yellowish grey
Bedding & Sedimentary Structures	No visible original depositional texture. Diagenetic process gives structure of coarsely crystalline calcite filled pore spaces. Silica replacement rounded and segregated in the dolomite.
Dunham Classification	DOLOMITIZED PELOIDAL GRAINSTONE (The original rock is a peloidal grainstone, then altered through diagenetic process, replaced extensively by dolomite)
Main Grain Types	Non skeletal/ peloid and crystalline
Visible Porosity	Intercrystalline, vug (strongly leached) or cavern in some places, and peloidal moldic
Cement/Matrix	Calcite as cement and matrix
Accessory Minerals	Dolomite and silica minerals (probably Chalcedony)
Oil Stain	No
Genetic Classification of Carbonate Porosity (Ahr)	Hybrid 1 (hybrid of depositional and diagenetic processes)

Sample #	CR-6
Date Received	3/20/2008
Location	College Station, TX
Formation	Unknown



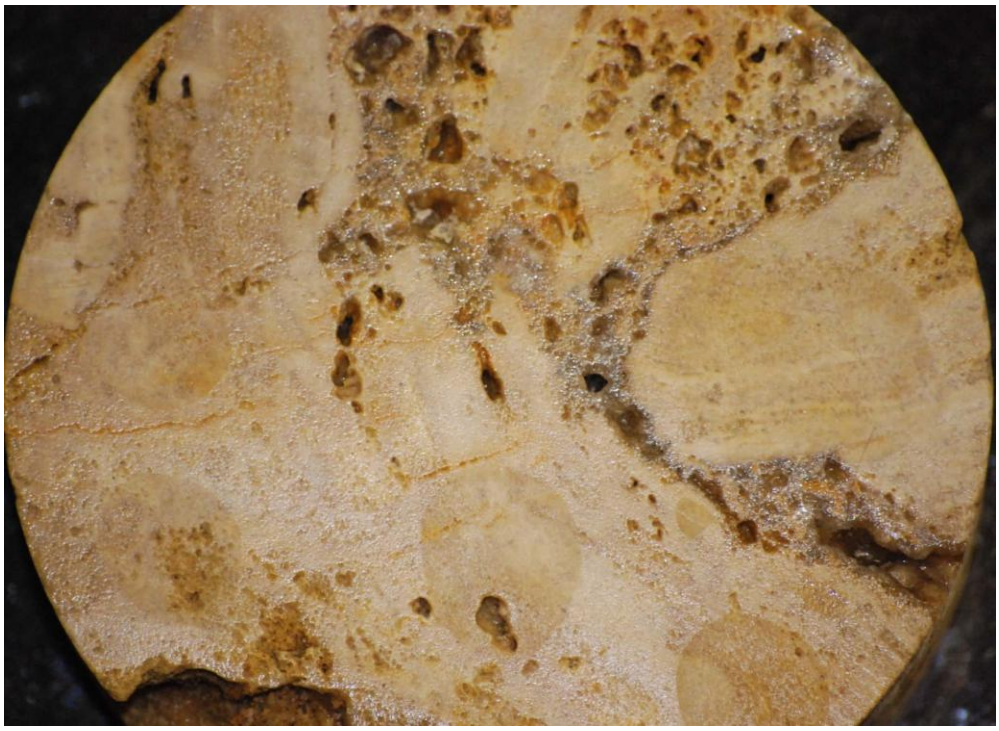
Rock Type	DOLOMITIC LIMESTONE
Color	Yellowish grey
Bedding & Sedimentary Structures	No visible original depositional texture. Diagenetic altered and completely recrystallized. Coarsely crystalline calcite filled pore spaces.
Dunham Classification	DOLOMITIZED PELOIDAL GRAINSTONE (The original rock is a peloidal grainstone, then altered through diagenetic process, replaced extensively by dolomite)
Main Grain Types	Non skeletal/ peloid (oolite?), and crystalline
Visible Porosity	Intercrystalline, vug (strongly leached), and peloidal moldic
Cement/Matrix	Calcite as cement and matrix
Accessory Minerals	Dolomite
Oil Stain	No
Genetic Classification of Carbonate Porosity (Ahr)	Hybrid 1 (hybrid of depositional and diagenetic processes)

Sample #	CR-7
Date Received	3/20/2008
Location	College Station, TX
Formation	Unknown



Rock Type	DOLOMITIC LIMESTONE
Color	Yellowish grey
Bedding & Sedimentary Structures	No visible original depositional texture. Diagenetic altered and completely recrystallized.
Dunham Classification	DOLOMITIZED PELOIDAL GRAINSTONE (The original rock is a peloidal grainstone, then altered through diagenetic process, replaced extensively by dolomite)
Main Grain Types	Non skeletal/ peloid, and crystalline
Visible Porosity	Intercrystalline, vug (strongly leached), and peloidal moldic
Cement/Matrix	Calcite as cement and matrix
Accessory Minerals	Dolomite
Oil Stain	No
Genetic Classification of Carbonate Porosity (Ahr)	Hybrid 1 (hybrid of depositional and diagenetic processes)

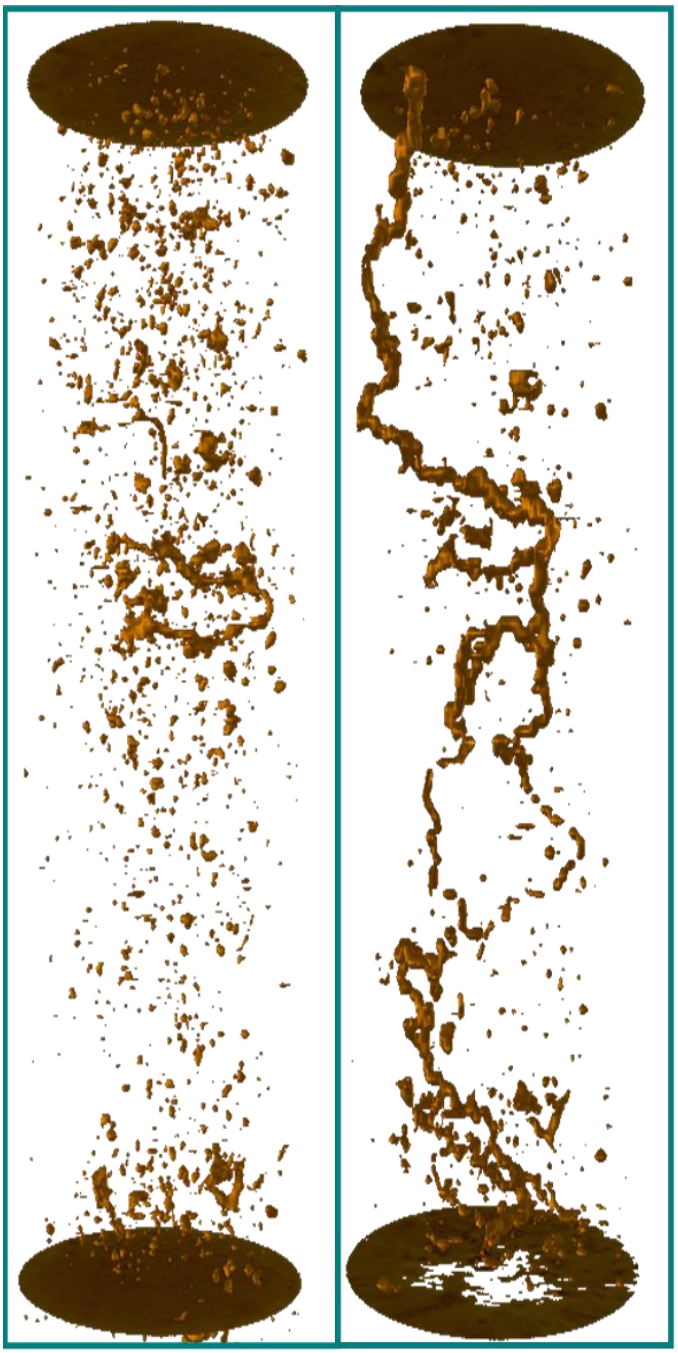
Sample #	CR-8
Date Received	3/20/2008
Location	College Station, TX
Formation	Unknown



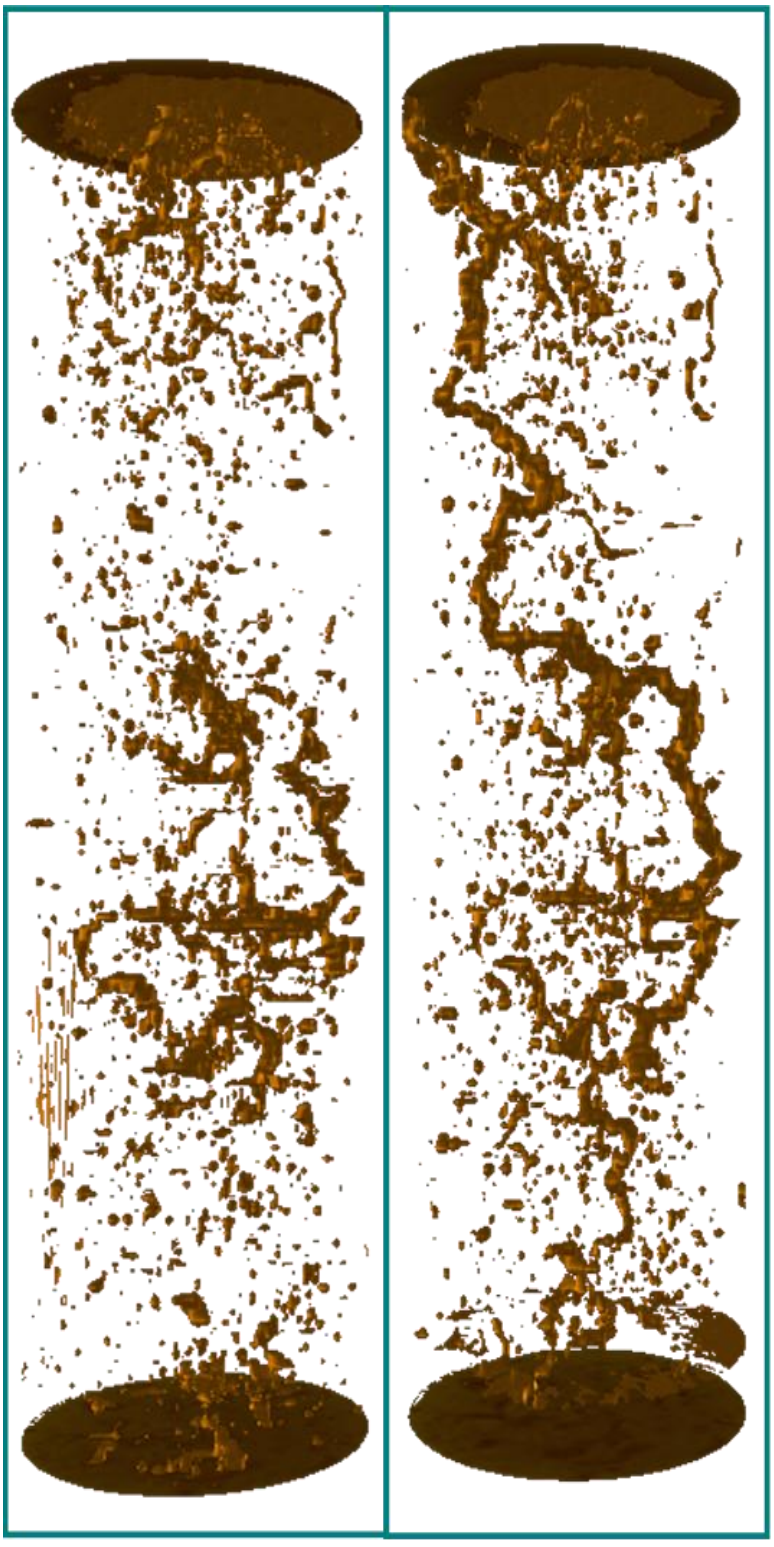
Rock Type	DOLOMITIC LIMESTONE
Color	Yellowish grey
Bedding & Sedimentary Structures	Even bedding, with distinctive banded layers: one section is dolomitized and the other is extensively leached. Coarsely crystalline calcite filled pore spaces.
Dunham Classification	DOLOMITIZED PELOIDAL GRAINSTONE (The original rock is a peloidal grainstone then altered by diagenetic process, replaced extensively by dolomite)
Main Grain Types	Non skeletal/ peloid, and crystalline
Visible Porosity	Intercrystalline, vug (strongly leached), peloidal moldic, and fenestral fabric . Most of porosity was occluded by a coarse meshwork of calcite crystal laths.
Cement/Matrix	Calcite as cement and matrix
Accessory Minerals	Dolomite
Oil Stain	No
Genetic Classification of Carbonate Porosity (Ahr)	Hybrid 1 (hybrid of depositional and diagenetic processes)

APPENDIX D

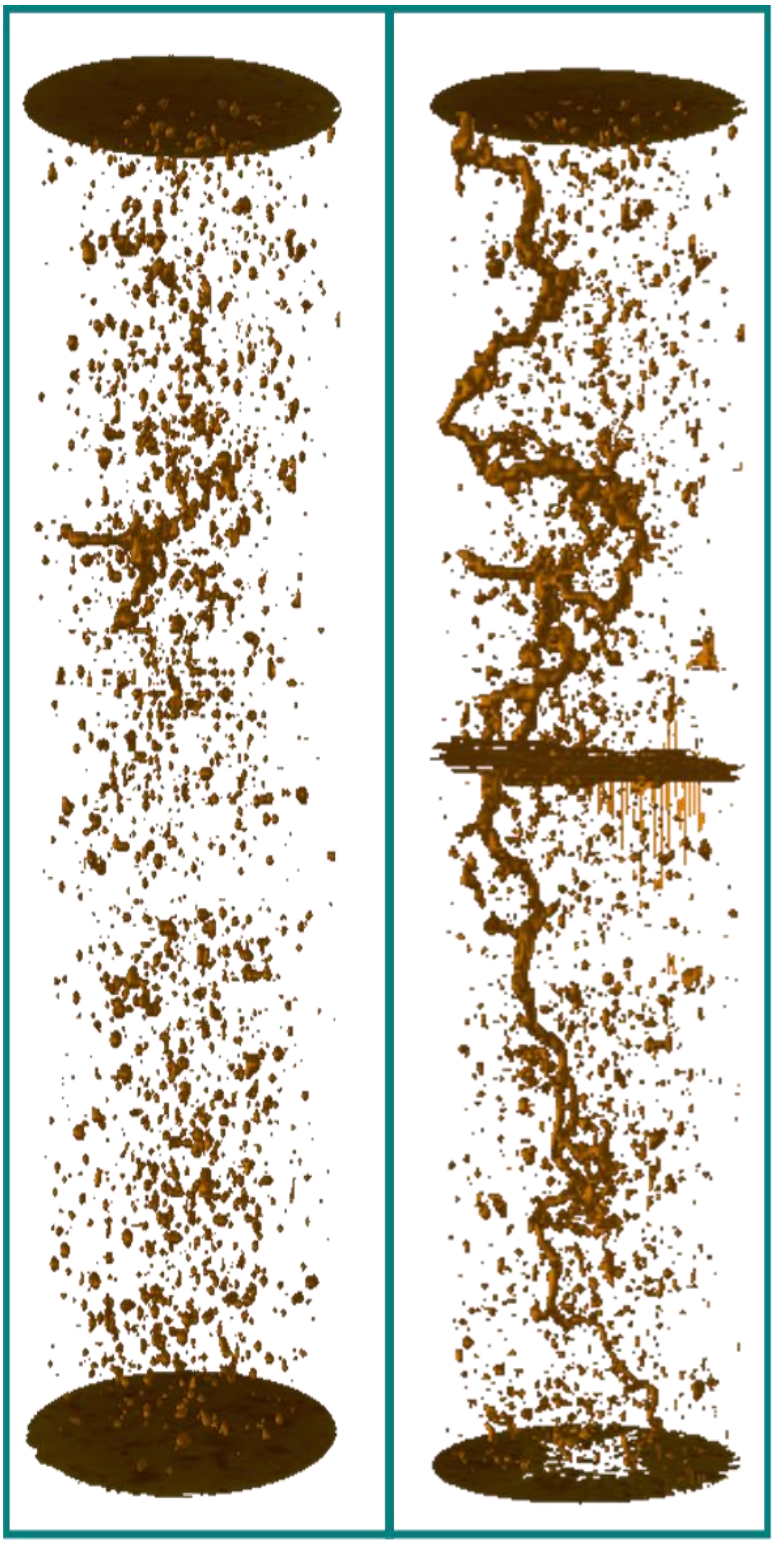
CT SCAN IMAGES



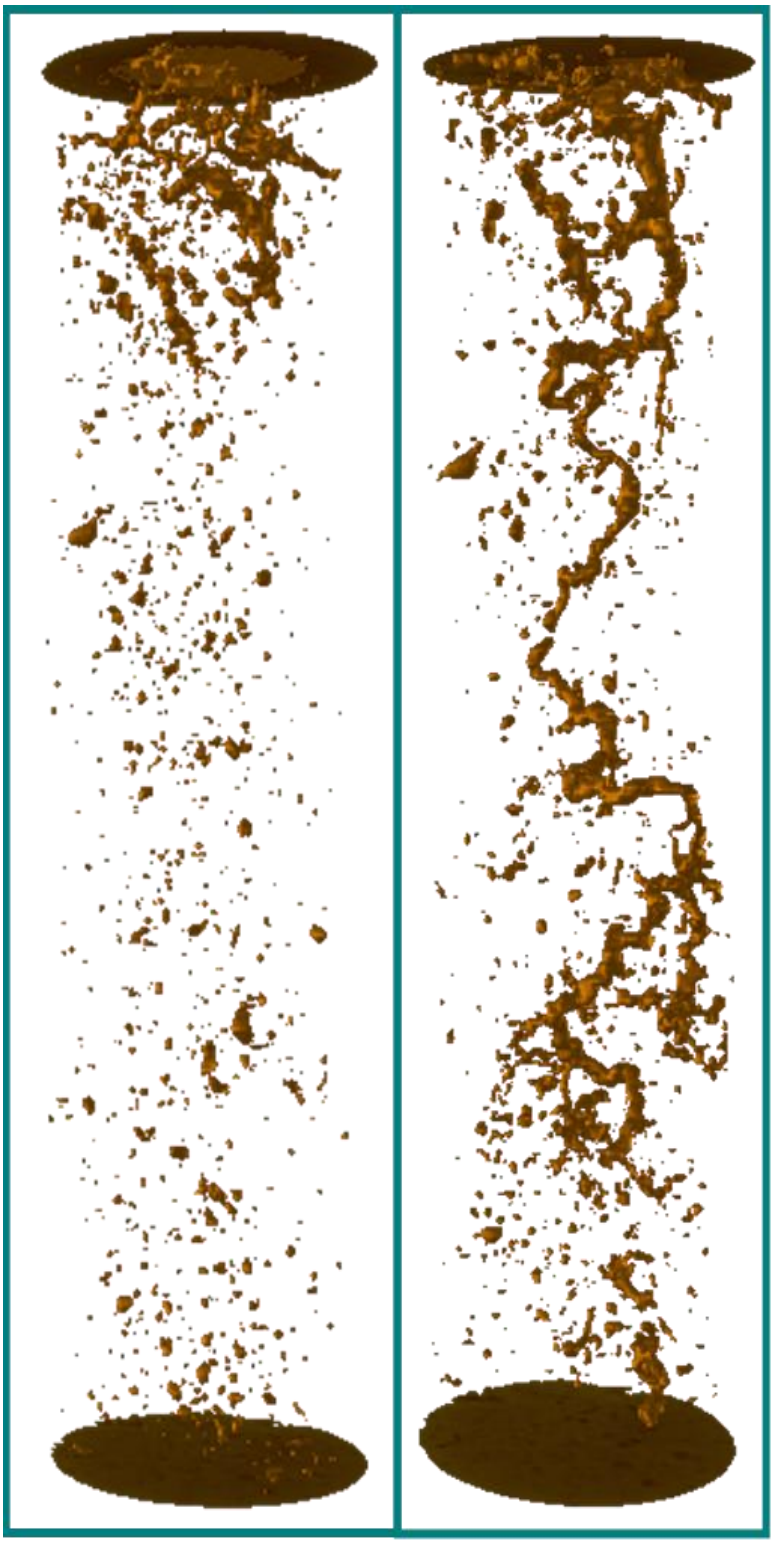
Before and after CT images for BC1



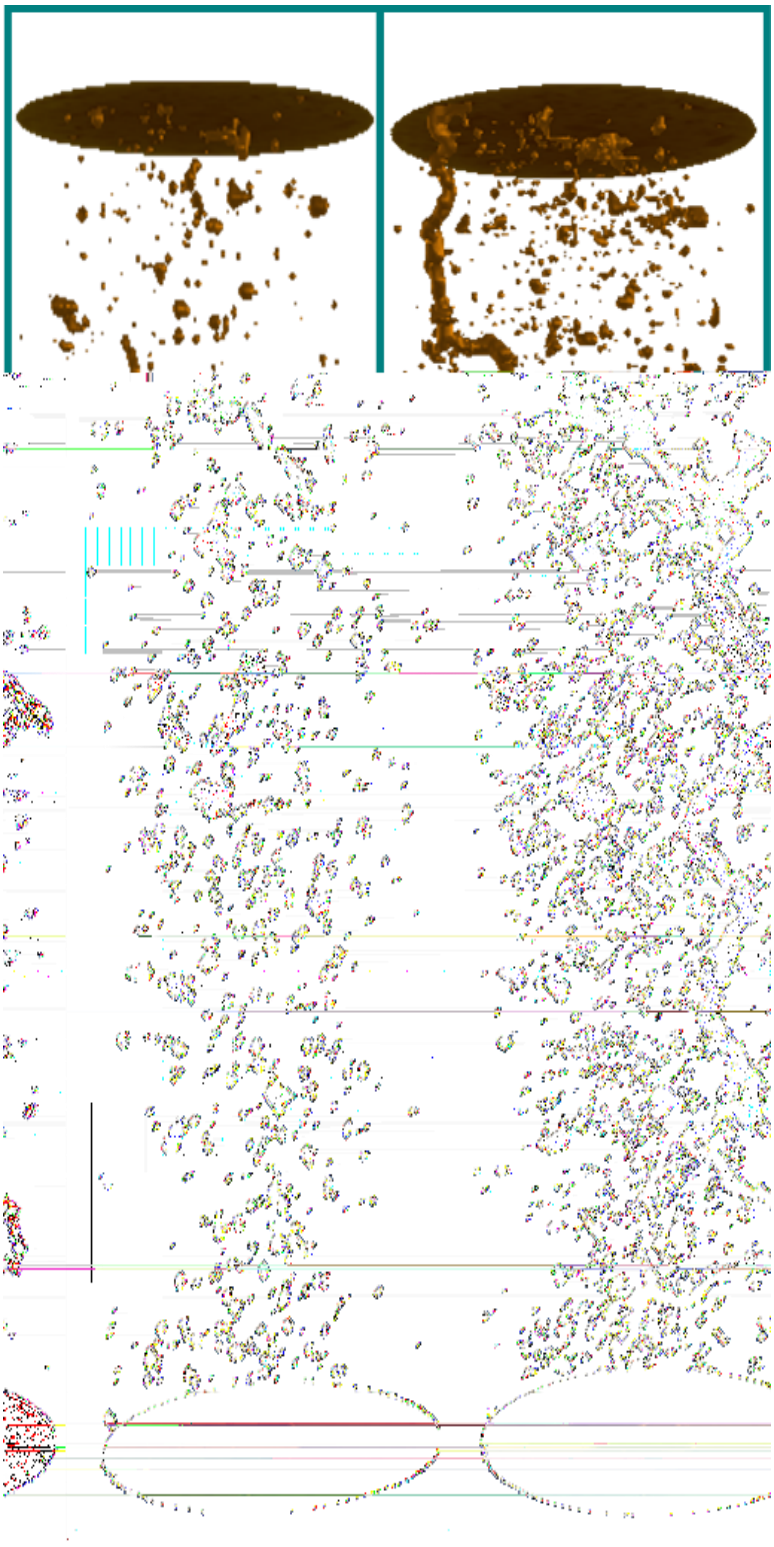
Before and after CT images for BC2



Before and after CT images for BC3

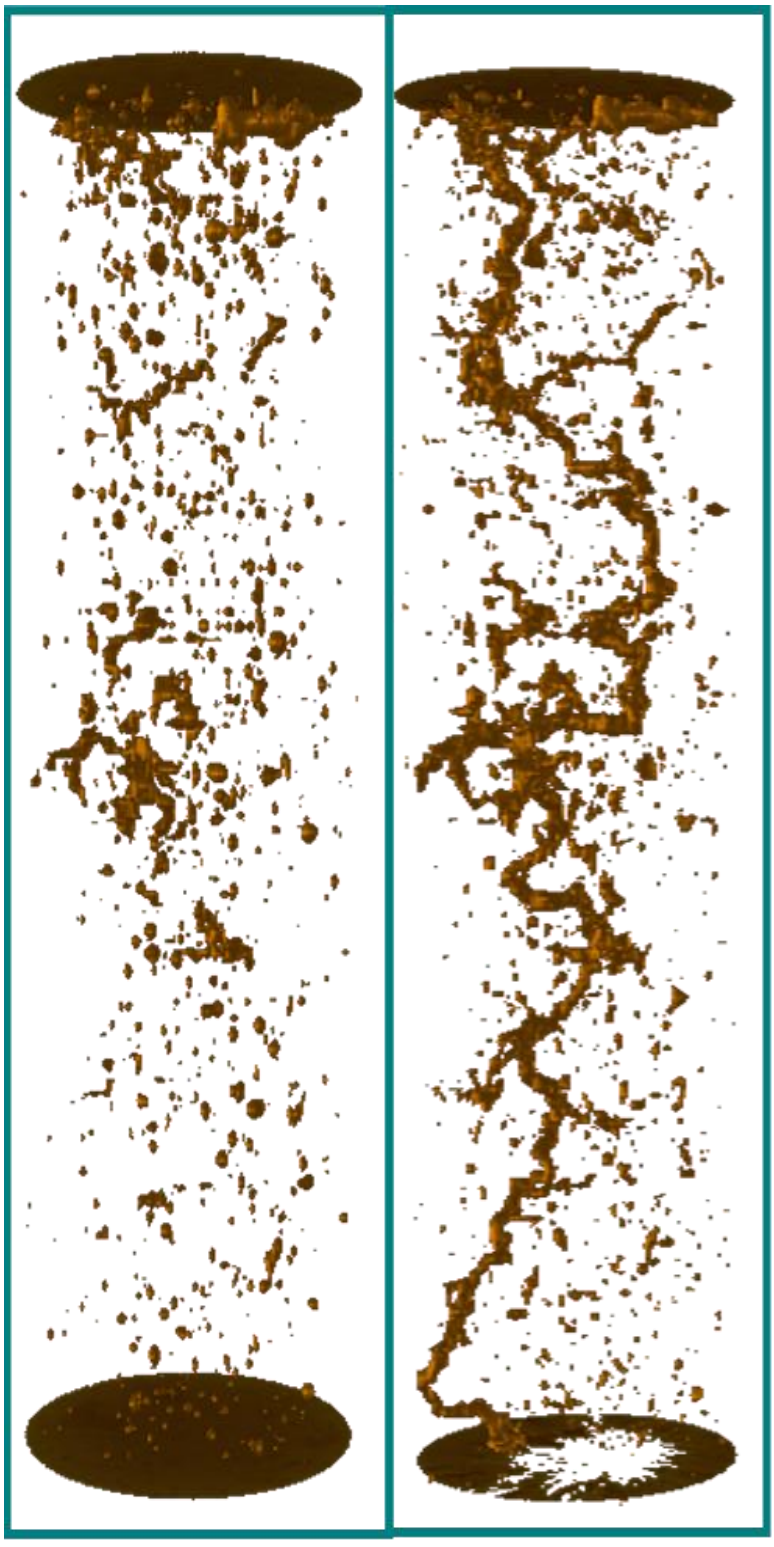


Before and after CT images for BC4

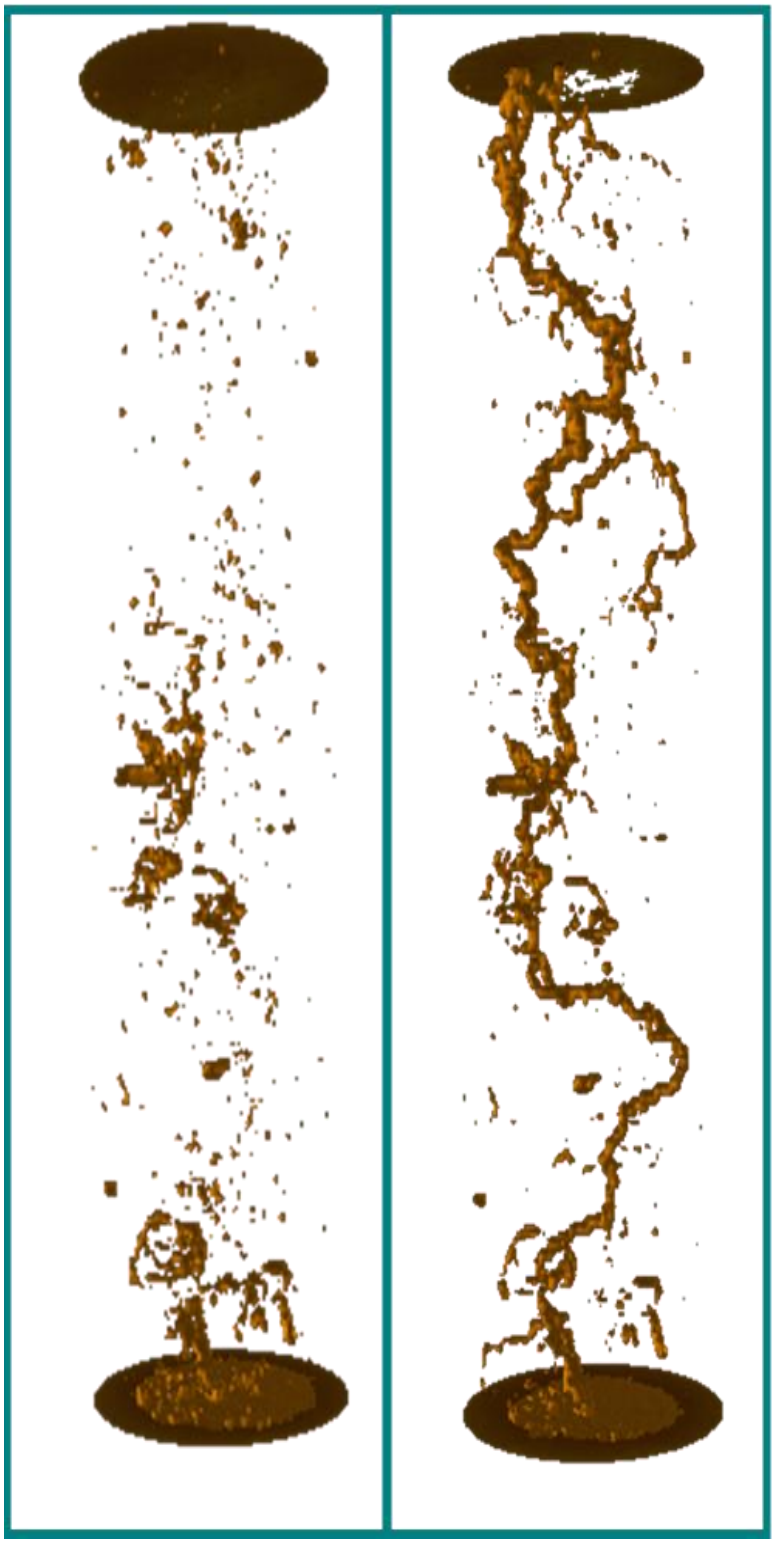


Before and after CT images for BC5

Before and after CT images for BC6



Before and after CT images for BC7

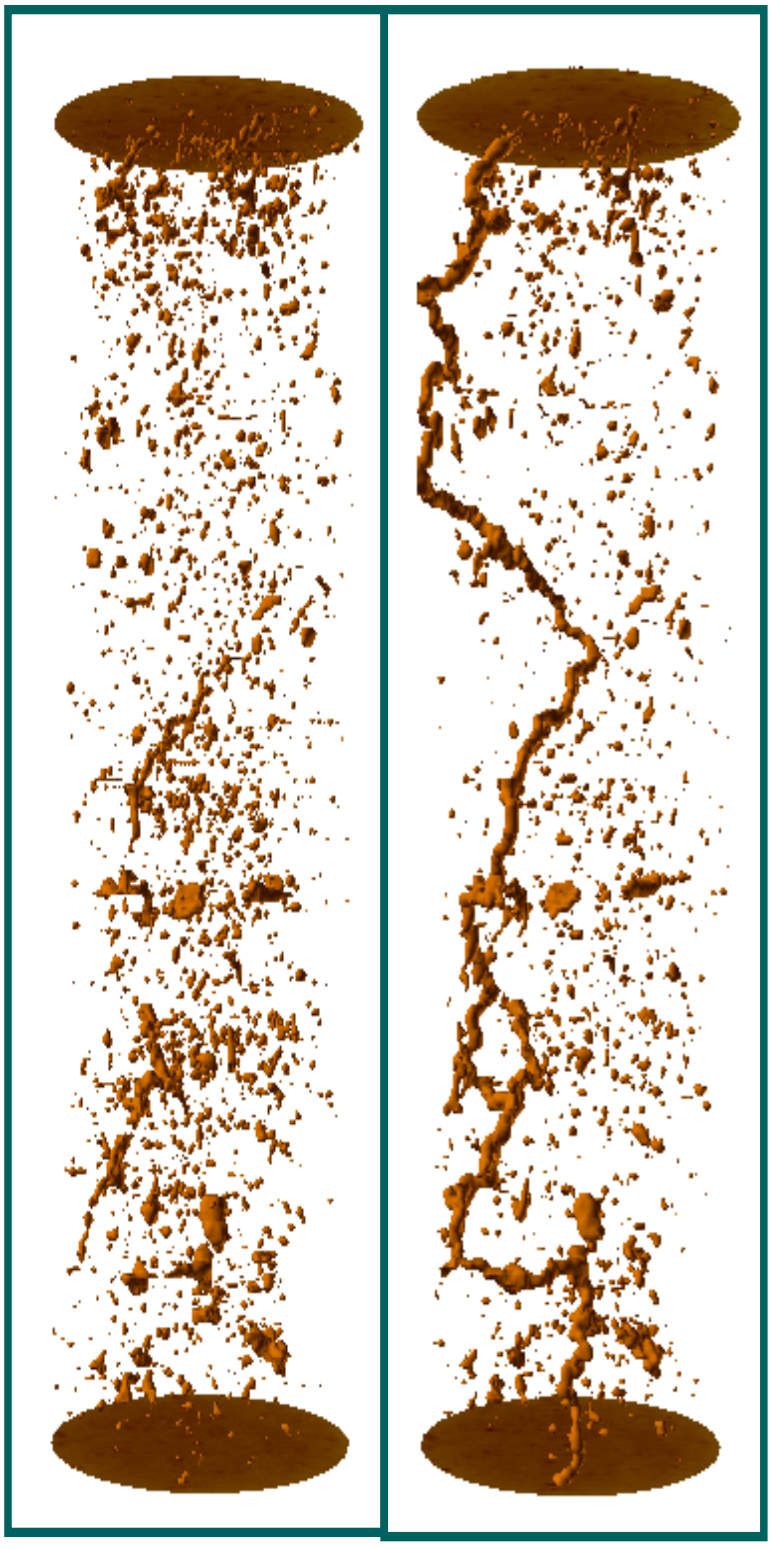


Before and after CT images for BC10

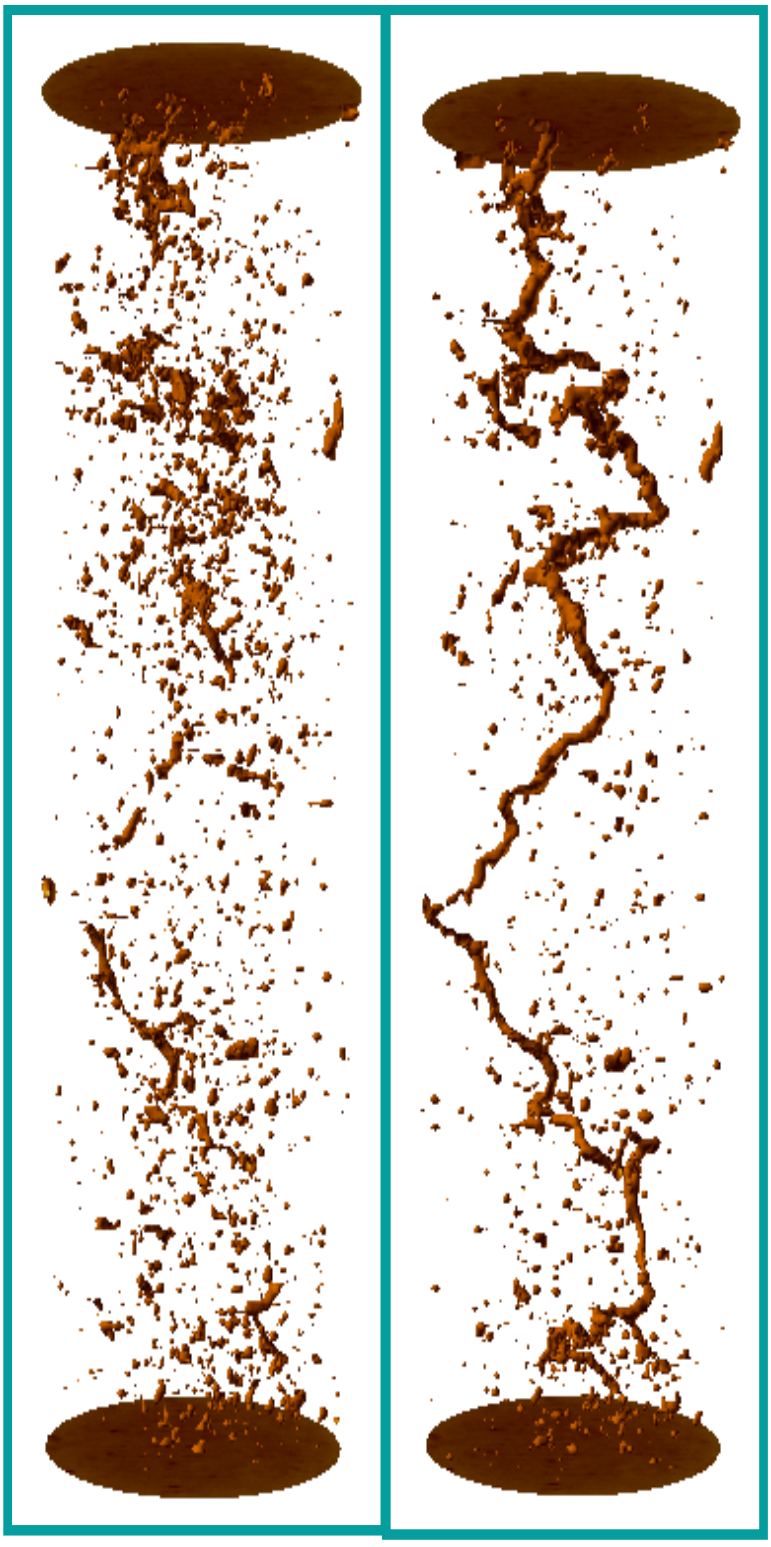
Before and after CT images for BC12

Before and after CT images for BC13

Before and after CT images for BC14



Before and after CT images for BC21



Before and after CT images for BC22

VITA

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